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Technology Evaluation Report: SITE Program Demonstration Test Soliditech, Inc., Solidification/Stabilization Process. Volume 1

PRC Environmental Management, Inc., Chicago, IL

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Cincinnati OH 45268

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SITE Program Demonstration Test Soliditech, Inc. Solidification/Stabilization Process

Volume I

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Technology Evaluation Report:

SITE Program Demonstration Test
Soliditech, Inc.
Solidification/Stabilization Process

Volume I

Risk Reduction Engineering Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268

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NOTICE

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16. ABSTRACT

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The technical criteria used to evaluate the effectiveness of the Soliditech process were contaminant mobility, based upon leaching and permeability tests; and the structural integrity of the solidified material, based upon physical and morphological tests.

The treated wastes had significant structural integrity, low permeability, and higher bulk density than the untreated wastes. pH values of the treated wastes were highly influenced by alkalinity of the portland cement added during treatment. Treatment resulted in significantly reduced concentrations of arsenic, lead, and zinc in (1) extracts from TCLP, EP Toxicity, and BET procedures; and (2) leachates from intact cast cylinders subjected to ANS 16.1 and WILT procedures. PCBs could not be detected in any extracts or leachates from treated wastes.

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FOREWORD

The Superfund Innovative Technology Evaluation (SITE) Program was authorized in the 1986 Superfund amendments. The program is a joint effort between EPA's Office of Research and Development (ORD) and Office of Solid Waste and Emergency Response (OSWER). The purpose of the program is to assist the development of hazardous waste treatment technologies necessary to implement new cleanup standards which require greater reliance on permanent remedies. This is accomplished through technology demonstrations which are designed to provide engineering and cost data on selected technologies.

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This project is a field demonstration under the SITE program and designed to analyze the Soliditech, Inc. solidification/stabilization technology. The technology demonstration took place at a former oil recycling facility in Morganville, New Jersey. The demonstration effort was directed at obtaining information on the performance and cost of the technology for assessing its use at this as well as other uncontrolled hazardous waste sites. Documentation will consist of two reports: (1) a Technology Evaluation Report that describes the field activities and laboratory results; and (2) an Applications Analysis Report that provides an interpretation of the data, and discusses the potential applicability of the technology.

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E. Timothy Oppelt, Director
Risk Reduction Engineering Laboratory

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The primary objective of the Soliditech, Inc., solidification/stabilization demonstration was to develop reliable performance and cost information. The demonstration took place at the Imperial Oil Company/Champion Chemical Company Superfund site in Morganville, New Jersey. Contamination at this site includes PCBs, various metals, and oil and grease. The Soliditech process mixes the waste material with Urrichem, a proprietary reagent; proprietary additives; pozzolanic materials or cement (cement was used for the demonstration); and water; in an open-top concrete mixer.

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VOLUME I

TABLE OF CONTENTS

Section Page

NOTICE ii

FOREWORD iii

ABSTRACT iv

ABBREVIATIONS xii

CONVERSION OF CUSTOMARY UNITS TO METRIC UNITS xiv

ACKNOWLEDGEMENT xv

1.0 EXECUTIVE SUMMARY 1

- 1.1 INTRODUCTION 1
- 1.2 PROCESS DESCRIPTION 2
- 1.3 SAMPLING AND ANALYSIS PROGRAM 3
- 1.4 FIELD OBSERVATIONS 4
- 1.5 ANALYTICAL RESULTS 4

2.0 INTRODUCTION 8

- 2.1 BACKGROUND 8
  - 2.1.1 SITE Program 8
  - 2.1.2 Soliditech Technology 9
  - 2.1.3 Site Background 9
- 2.2 TECHNOLOGY DEMONSTRATION PROGRAM OBJECTIVES 11
- 2.3 TECHNOLOGY EVALUATION CRITERIA 11
- 2.4 DESCRIPTION OF OPERATIONS 14
- 2.5 PROJECT ORGANIZATION 15

3.0 CONCLUSIONS 16

- 3.1 SUMMARY OF PERFORMANCE DATA 16
- 3.2 SUMMARY OF MAJOR PROBLEMS 20
- 3.3 ADVANTAGES AND LIMITATIONS OF THE SOLIDITECH PROCESS ... 20
  - 3.3.1 Advantages of the Soliditech Process 20
  - 3.3.2 Limitations of the Soliditech Process 27

4.0 SOLIDITECH PROCESS 29

- 4.1 PROCESS DESCRIPTION 29
- 4.2 PROCESS SCHEMATIC 29
- 4.3 EQUIPMENT SPECIFICATIONS 32

TABLE OF CONTENTS

Section Page

5.0 DEMONSTRATION PROCEDURES . . . 35

- 5.1 SITE DESCRIPTION . . . 35
- 5.2 WASTE CHARACTERISTICS . . . 36
- 5.3 DEMONSTRATION PREPARATION . . . 38
- 5.4 SAMPLING PROGRAM . . . 38
  - 5.4.1 Waste Material Collection Locations . . . 38
  - 5.4.2 Sampling Schedule . . . 38
    - 5.4.2.1 Pretreatment Sampling Methods and Types . . . 43
    - 5.4.2.2 Reagent Mix Sampling Methods and Types . . . 50
    - 5.4.2.3 Post-Treatment Sampling Methods and Types . . . 50
- 5.5 PHYSICAL TESTS . . . 51
  - 5.5.1 On-Site Tests . . . 51
  - 5.5.2 Laboratory Tests . . . 51
- 5.6 CHEMICAL TESTS . . . 53
- 5.7 LEACHING TESTS . . . 59
- 5.8 QUALITY ASSURANCE AND QUALITY CONTROL SUMMARY . . . 61

6.0 FIELD ACTIVITIES . . . 63

- 6.1 WASTE MATERIAL COLLECTION . . . 63
  - 6.1.1 Summary of Operations . . . 63
  - 6.1.2 Deviations from the Demonstration Plan . . . 66
- 6.2 WASTE TREATMENT . . . 66
  - 6.2.1 Summary of Test Runs . . . 67
  - 6.2.2 Deviations from the Demonstration Plan . . . 69
- 6.3 SAMPLING FOR PROCESS EVALUATION . . . 70
  - 6.3.1 Pretreatment Sampling Procedures . . . 70
  - 6.3.2 Post-Treatment Sampling Procedures . . . 70
  - 6.3.3 Sampling Deviations . . . 75

7.0 PERFORMANCE DATA AND EVALUATION . . . 76

- 7.1 PHYSICAL TESTS . . . 76
  - 7.1.1 Bulk Density . . . 76
  - 7.1.2 Water Content . . . 76
  - 7.1.3 Particle Size Distribution . . . 78

TABLE OF CONTENTS

Section Page

7.1.4 Permeability 78
7.1.5 Unconfined Compressive Strength 78
7.1.6 Wet/Dry Weathering Test 78
7.1.7 Freeze/Thaw Weathering Test 79
7.1.8 Loss on Ignition 78
7.1.9 On-Site Tests 79

7.2 CHEMICAL TESTS 79

7.2.1 pH 80
7.2.2 Eh 80
7.2.3 Metals 80
7.2.4 Polychlorinated Biphenyls 84
7.2.5 Volatile Organic Compounds 84
7.2.6 Semivolatile Organic Compounds 85
7.2.7 Oil and Grease 86
7.2.8 Acid Neutralization Capacity/Neutralization Potential 86

7.3 LEACHING TESTS 86

7.3.1 TCLP 87

7.3.1.1 Reagent Mix 87
7.3.1.2 Filter Cake 87
7.3.1.3 Filter Cake/Oily Sludge 89
7.3.1.4 Off-Site Area One 90

7.3.2 EP Toxicity 91

7.3.2.1 Reagent Mix 91
7.3.2.2 Filter Cake 91
7.3.2.3 Filter Cake/Oily Sludge 93
7.3.2.4 Off-Site Area One 93

7.3.3 BET 94

7.3.3.1 Reagent Mix 94
7.3.3.2 Filter Cake 94
7.3.3.3 Filter Cake/Oily Sludge 99
7.3.3.4 Off-Site Area One 100

7.3.4 ANS 16.1 101

7.3.4.1 Filter Cake 101
7.3.4.2 Filter Cake/Oily Sludge 105
7.3.4.3 Off-Site Area One 105
7.4 MORPHOLOGICAL TESTS 105
7.5 LONG-TERM TESTS 107

7.5.1 Waste Interface Leaching Test 107
7.5.2 TCLP and EP Extracts of Solidified Waste 108

TABLE OF CONTENTS

Section | Page

7.5.3 PHYSICAL STABILITY OF TREATED WASTE MONOLITHS 110

7.5.3.1 SURFACE SPALLING 110
7.5.3.2 GRAIN EXFOLIATION 111
7.5.3.3 CRACK AND FISSURE DEVELOPMENT 111
7.5.3.4 OXIDATIVE DISCOLORATION 111
7.5.3.5 SALT EFFLORESCENCE 112
7.5.3.6 PORE CHARACTERIZATION 112

7.6 MATERIALS BALANCE 112

8.0 COST OF DEMONSTRATION 116

8.1 U.S. EPA SITE CONTRACTOR COSTS 116

8.1.1 Phase I: Planning Stage 116
8.1.2 Phase II: Demonstration Stage 117

8.2 DEVELOPER (SOLIDITECH) COSTS 117

REFERENCES 119

Appendices

A LABORATORY DATA FROM THE TECHNOLOGY DEMONSTRATION

B LABORATORY QA/QC RESULTS

C LABORATORY DATA FROM THE TREATABILITY STUDY

D RESULTS OF FIELD EXPERIMENTS

E PRC TRIP REPORT

F SOLIDITECH DOCUMENTATION OF FIELD REPORTS

G PRELIMINARY WILT RESULTS

LIST OF TABLES

Table		Page
1	PHYSICAL PROPERTIES	5
2	CHEMICAL PROPERTIES	6
3	TCLP AND EP TOXICITY LEACHATE RESULTS FOR FILTER CAKE WASTE	21
4	TCLP AND EP TOXICITY LEACHATE RESULTS FOR FILTER CAKE/OILY SLUDGE MIXTURE	22
5	TCLP AND EP TOXICITY LEACHATE RESULTS FOR OFF-SITE AREA ONE WASTE	23
6	BET EXTRACT RESULTS FOR FILTER CAKE WASTE	24
7	BET EXTRACT FOR FILTER CAKE/OILY SLUDGE MIXTURE	25
8	BET EXTRACT FOR OFF-SITE AREA ONE WASTE	26
9	SOLIDITECH TREATMENT FORMULATIONS	30
10	SAMPLING REQUIREMENTS	39
11	STANDARD METHODS AND PROCEDURES OF SAMPLE ANALYSIS	44
12	DIGESTION AND ANALYTICAL TECHNIQUES FOR ELEMENTAL ANALYSIS	58
13	SOLIDITECH DEMONSTRATION CHRONOLOGY	64
14	CHEMICAL AND PHYSICAL ANALYSES OF UNTREATED AND TREATED WASTE	77
15	CHEMICAL ANALYSIS OF SAND	81
16	CHEMICAL ANALYSIS OF PREDEMONSTRATION BLANK	82
17	CHEMICAL ANALYSES OF TCLP EXTRACT FROM UNTREATED AND TREATED WASTE MATERIALS	88
18	CHEMICAL ANALYSES OF EP EXTRACT FROM UNTREATED AND TREATED WASTE	92
19	CHEMICAL ANALYSES OF BET EXTRACT FROM UNTREATED AND TREATED FILTER CAKE WASTE	95
20	CHEMICAL ANALYSES OF BET EXTRACT FROM UNTREATED AND TREATED FILTER CAKE/OILY SLUDGE MIXTURE	96
21	CHEMICAL ANALYSES OF BET EXTRACT FROM UNTREATED AND TREATED OFF-SITE AREA ONE WASTE	97

LIST OF TABLES

Table Page

22 CHEMICAL ANALYSES OF BET EXTRACT FROM REAGENT MIX 98

23 CHEMICAL ANALYSES OF ANS 16.1 LEACHATE FROM TREATED FILTER CAKE WASTE 102

24 CHEMICAL ANALYSES OF ANS 16.1 LEACHATE FROM TREATED FILTER CAKE/OILY SLUDGE MIXTURE 103

25 CHEMICAL ANALYSES OF ANS 16.1 LEACHATE FROM TREATED OFF-SITE AREA ONE WASTE 104

26 WILT TEST RESULTS THROUGH WEEK 16 109

27 MATERIALS BALANCE 113

28 OBSERVED VOLUME AND WEIGHT OF TREATED MATERIAL 114

LIST OF FIGURES

Figure — Page

1 SITE LOCATION MAP 10

2 IMPERIAL OIL COMPANY/CHAMPION CHEMICALS SITE WASTE PILE AND ABANDONED STORAGE TANK 12

3 IMPERIAL OIL COMPANY/CHAMPION CHEMICALS SITE OFF-SITE AREAS 13

4 SOLIDITECH PROCESS SCHEMATIC 31

5 WASTE CONTAINED IN 10-CUBIC-YARD SOLIDITECH MIXER 65

6 DISCHARGING OF TREATED WASTE INTO PLYWOOD FORMS 68

7 SAMPLES OF TREATED WASTE IN CYLINDRICAL MOLDS 72

8 LARGE MONOLITHS OF TREATED WASTE BEING PREPARED FOR LONG-TERM STUDY 73

9 STACKING DIAGRAM OF THE TREATED WASTE MONOLITHS 74

ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
ANS-16.1	Leach test used by the American Nuclear Society
ASA	American Society of Agronomy
ASTM	American Society for Testing and Materials
BET	Batch Extraction Test
C	Celsius
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
cm	centimeter
CVAAS	Cold Vapor Atomic Absorption Spectroscopy
EP	Extraction Procedure Toxicity Test
EPA	Environmental Protection Agency
Eh	Oxidation/Reduction Potential
F	Fahrenheit
g	gram
gal	gallon
GC/ECD	Gas Chromatography/Electron Capture Detection
GC/MS	Gas Chromatography/Mass Spectrometer
GFAAS	Graphite Furnace Atomic Absorption Spectroscopy
HDPE	High-Density Polyethylene
hr	hour
ICP	Inductively Coupled Argon Plasma Spectroscopy
Kg	Kilogram
kwhr	kilowatt/hour
L	liter
lb	pound
M	Molarity
mg	milligram
mg/Kg	milligrams per Kilogram
mg/L	milligrams per Liter
min	minute
mL	milliliter
mm	millimeter
mv	millivolts
N	Normality
NC	Not Calculated
ND	Not Detected
NJ DEP	New Jersey Department of Environmental Protection
NPL	National Priority List
ORD	Office of Research and Development
OSWER	Office of Solid Waste and Emergency Response
PCB	Polychlorinated Biphenyl
PRC	Planning Research Corporation Environmental Management, Inc.
psi	pounds per square inch
PVC	Polyvinyl Chloride
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation and Feasibility Study
RPM	Revolutions Per Minute
RSD	Relative Standard Deviation
SARA	Superfund Amendments and Reauthorization Act

ABBREVIATIONS (Continued)

sec	second
SEM	Scanning Electron Microscope
SITE	Superfund Innovative Technology Evaluation
S/L	Solid to Liquid Ratio
SVOC	Semivolatile Organic Compound
TCLP	Toxicity Characteristics Leaching Procedure
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TMSWC	Test Methods for Solidified Waste Characterization
TSCA	Toxic Substances Control Act
UCS	Unconfined Compressive Strength
μg	micrograms
μg/L	micrograms per Liter
μm	micrometer
μmho	units of conductance
VOC	Volatile Organic Compound
WES	Waterways Experiment Station
WILT	Waste Interface Leaching Test
wt	weight
XRD	X-Ray Diffraction
yd	yard
ZHE	Zero Headspace Extractor

CONVERSION OF CUSTOMARY UNITS TO METRIC UNITS

Length

inches X 2.54 = centimeters

inches X 0.0254 = meters

feet X 0.3048 = meters

Volume

gallons X 3.785 = liters

cubic yards X 0.7646 = cubic meters

Weight

pounds X 0.4536 = kilograms

short tons X 0.9072 = metric tons

Temperature

5/9 X (° Farenheit - 32) = ° Celsius

Note: 1000 liters = 1 cubic meter

1000 kilograms = 1 metric ton

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1.0 EXECUTIVE SUMMARY

1.1 INTRODUCTION

In response to the Superfund Amendments and Reauthorization Act of 1986 (SARA), U.S. EPA has established a formal program to accelerate the development, demonstration, and use of new or innovative technologies that offer permanent, long-term cleanup solutions at Superfund sites. This program is called the Superfund Innovative Technology Evaluation or SITE program, and is administered by the Office of Solid Waste and Emergency Response (OSWER) and the Office of Research and Development (ORD).

The SITE program has four primary goals:

- **To identify and remove impediments to the development and commercial use of alternate technologies.**
- **To conduct a demonstration of the more promising innovative technologies to establish reliable performance and cost information for site characterization and cleanup decision making.**
- **To develop procedures and policies that encourage the selection of available alternative treatment remedies at Superfund sites as well as other waste sites and commercial facilities.**
- **To structure a development program that nurtures emerging technologies.**

In January 1987, U.S. EPA solicited proposals from approximately 400 developers of innovative waste treatment technologies who had expressed an interest in becoming involved in the SITE program. U.S. EPA received 29 proposals by the March 13, 1987 due date. In September 1987, EPA selected 10 technologies for inclusion in the SITE demonstration program (002). One of these was the solidification/stabilization process developed by Soliditech, Inc., of Houston, Texas.

The Imperial Oil Company/Champion Chemical Company Superfund site in Morganville, New Jersey was chosen for the Soliditech demonstration. The chemicals of concern at this site include metals such as arsenic, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, thallium, and zinc; and various organic chemicals including polychlorinated biphenyls (PCBs) and petroleum hydrocarbons. Contamination is present in the soil, in a waste filter cake pile, and in an abandoned storage tank, as well as in the ground water. Samples of contaminated material from the soil, the waste filter cake pile, and the abandoned storage tank were treated during the demonstration.

The four major objectives identified for the Soliditech SITE demonstration were to:

- Determine the effectiveness of the technology to solidify and stabilize waste materials found at the site.
- Determine whether the solidified material maintains its structural properties and stabilization effectiveness over a five-year period.
- Determine the volume and mass change of the solidified material due to addition of pozzolans, water, reagent, and other proprietary additives.
- Develop reliable capital and operating costs for the technology that can be used in the Superfund decision-making process.

To meet these objectives, a SITE Demonstration Plan was prepared (PRC, 1988b). This plan detailed all sampling and analysis to be performed during the Soliditech demonstration. Analytical tests were performed on samples of untreated as well as solidified waste material collected during the demonstration. The results will be used to evaluate the effectiveness of the treatment process and the structural properties of the resulting solidified material. Soliditech personnel maintained operating logs to determine the capital and operating expenses associated with the demonstration. Soliditech and U.S. EPA personnel maintained field logs of the volume and weight of all ingredients for each test run, as well as the volume and weight of all treated material. These data are presented in this report and have been analyzed in view of the above objectives.

1.2 PROCESS DESCRIPTION

Soliditech, Inc. claims that its solidification/stabilization process chemically and physically immobilizes hazardous constituents in waste material. This immobilization occurs by one or more of the following processes: encapsulation, adsorption, and incorporation into the crystalline structure of the solidified material. The Soliditech process uses a proprietary reagent (Urrichem); proprietary additives; pozzolans (such as fly ash), kiln dust, or cement; and water to solidify solids and sludge containing organic and inorganic chemicals typically found at hazardous waste sites.

The chemical reagent and proprietary additives are mixed in a batch process with the waste material, water, and the pozzolanic materials to solidify the waste materials by chemical and physical processes. Once thoroughly mixed, the treated waste is discharged from the mixer and allowed to cure. The final product is claimed to be a monolithic material with measurable structural strength and significantly reduced leaching potential.

During the demonstration, waste materials from three site locations were collected, screened, mixed, and treated by the Soliditech process. In addition, clean sand was treated as a control. The treated material was placed in small, cylindrical molds for chemical and physical testing, and in large 1-cubic yard plywood forms for long-term monitoring, and allowed to cure for 28 days. After curing, the small cast sample cylinders were shipped to the analytical laboratories where they were analyzed. After the plywood sides of the large forms were removed, the resulting treated waste monoliths were placed in an enclosed on-site storage area for long-term monitoring.

1.3 SAMPLING AND ANALYSIS PROGRAM

Samples of waste material from four areas of the site were collected six months prior to the demonstration (PRC, 1988a). A portion of this material was supplied to Soliditech for testing to determine the suitability of the waste for treatment. Both raw and treated samples of the waste material were chemically analyzed to determine levels of contamination in the four areas, to assess the effectiveness of the Soliditech bench scale tests, and to allow the analytical laboratory to assess possible analytical problems. Additionally, Toxicity Characteristics Leaching Procedure (TCLP) tests were performed on the samples of raw and treated waste material and the leachate was chemically analyzed (Radian, 1988). Three areas containing three distinct waste types were chosen for treatment during the full-scale field demonstration.

During the SITE demonstration, pretreatment waste samples were collected for each test parameter from each of the three waste materials to be treated. These samples were analyzed for chemical constituents, physical characteristics, and ability to withstand leaching/extraction using both destructive and nondestructive techniques. The results allow a direct comparison of physical and chemical properties between the treated and untreated waste, and help determine the effectiveness of the treatment process.

A control run was performed to determine whether the Soliditech proprietary reagent and proprietary additives were contributing contamination to treated waste samples, and to set baseline values for some of the physical properties. Samples from the control run are referred to reagent mix samples. Clean sand was used as a surrogate waste material in the control run. These samples were analyzed for chemical constituents and physical characteristics.

Treated waste samples were collected immediately after each of the three waste treatment test runs and analyzed for chemical constituents, physical characteristics, and ability to withstand leaching/extraction. Samples for long-term testing were also collected. The long-term study includes a twelve-month, nondestructive leaching experiment, leaching by other leaching procedures at various times up to five years after treatment, and petrographic analysis and observation. These tests will help to assess the long-term stability of the treated material.

1.4 FIELD OBSERVATIONS

Several significant observations were made during the demonstration. These are summarized below.

A small amount of contaminated waste material was treated during the demonstration. For this small amount of material, mobilization and demobilization times were found to be disproportionately long. Additionally, special pre- and post-treatment procedures were followed to meet the demonstration objectives. During the remediation of a hazardous waste site, the time expended performing these activities would be insignificant compared to the actual waste treatment activities.

Measuring the exact weight or volume of wastes that were treated was difficult. This problem was largely due to the inability to accurately determine the weights of all ingredients. This should not be a problem in the field, when numerous batches and similar wastes are treated.

The adequacy of mixing of each batch of waste material was determined by the Soliditech operator, apparently based upon past experience rather than an objective standard. After curing, the solidified waste was observed to contain small amounts of unmixed material. Skilled operators or an objective standard are necessary to determine when waste is adequately mixed.

1.5 ANALYTICAL RESULTS

The analyses of the samples collected before, during, and after the Soliditech demonstration are summarized in Tables 1 and 2. The results are discussed below.

Pretreatment waste from the site consisted of contaminated soil, filter cake, and filter cake/oily sludge. These wastes contained 2.8 to 17 percent oil and grease with relatively low levels of other organic compounds. PCB (Aroclors 1242 and 1260) concentrations ranged from 28 to 43 mg/Kg in the pretreatment wastes. Lead concentrations ranged from 650 to 2500 mg/Kg in the pretreatment wastes.

TABLE 1

PHYSICAL PROPERTIES

	Filter Cake		Filter Cake/Oily Sludge Mixture		Off-Site Area One	
	Untreated	Treated[a]	Untreated	Treated[a]	Untreated	Treated[a]
Bulk Density (g/cm^3)	1.14	1.43	1.19	1.68	1.26	1.59
Permeability (cm/sec)	NA[b]	4.53×10^{-7}	NA	8.93×10^{-9}	NA	3.41×10^{-8}
Unconfined Compressive Strength (psi)	NA	390	NA	860	NA	680
Loss on Ignition (%)	54	41	70	34	36	34
Water Content (%)	28.7	21.0	58.1	14.7	23.5	12.6

Notes:

[a] Treated waste sampled after a 28-day curing period.

[b] NA = Not analyzed.

TABLE 2

CHEMICAL PROPERTIES

	Filter Cake				Filter Cake/Oily Sludge Mixture				Off-Site Area One			
Chemical Parameter(a)	Untreated Waste	Treated Waste(b)	Leachate from Untreated Waste(c)	Leachate from Treated Waste(c)	Untreated Waste	Treated Waste(b)	Leachate from Untreated Waste(c)	Leachate from Treated Waste(c)	Untreated Waste	Treated Waste(b)	Leachate from Untreated Waste(c)	Leachate from Treated Waste(c)
pH	3.4	11.8	4.6	10.8	3.6	12.0	4.8	11.6	7.9	12.0	5.1	11.5
VOCs(d)	ND(e)	ND	0.27(f)	ND	50(f)	ND	1.3(f)	ND	10	ND	0.87(f)	ND
SVOCs(g)	ND	36(f)	ND	1.2	63(f)	17(f)	0.38	0.97(f)	79(f)	16(f)	0.12(f)	0.32(f)
PCBs(h)	28	16	ND	ND	43	15	ND	ND	43	40	ND	ND
Oil and Grease	170,000	77,000	1.4	4.4	130,000	60,000	1.6	2.4	28,000	46,000	1.9	12
Arsenic	26	28	0.005	ND	14	40	0.014	ND	94	92	0.19	ND
Lead	2,200	680	4.3	0.002	2,500	850	5.4	0.014	650	480	0.55	0.012
Zinc	26	23	0.28	ND	150	54	1.3	ND	120	95	0.63	ND

Notes:

a Analyte concentration units for the untreated and treated waste are mg/Kg. Analyte concentration units for the leachate from untreated and treated waste are mg/L.

b Treated wastes were sampled after a 28-day curing period.

c Leachate values refer to results from TCLP test.

d VOCs = volatile organic compounds.

e ND = not detected.

f These values contain low levels of acetone, methylene chloride, various phthalates, or other analytes which are commonly attributed to sampling or analytical contamination.

g SVOCs = semivolatile organic compounds.

h PCBs = polychlorinated biphenyls.

The Soliditech solidification/stabilization process produced solidified waste with significant structural stability (UCS values ranging from 390 to 860 psi) and low permeability (10^{-9} to 10^{-7} cm/sec). Because of the cement in the Soliditech process, pH values of the solidified wastes ranged from 11.8 to 12.0. Lead concentrations ranged from 480 to 850 mg/Kg in the post-treatment wastes. PCB (Aroclors 1242 and 1260) concentrations ranged from approximately 15 to 41 mg/Kg in the post-treatment wastes. Oil and grease concentrations in the treated wastes ranged from 4.6 to 7.7 mg/Kg. Low concentrations of phenol (12 and 4.8 mg/Kg) and p-cresol (14 and 4.4 mg/Kg) were found in solidified filter cake and filter cake/oily waste samples.

The reagent mixture contained 20 mg/Kg of lead. PCBs, phenols, oil and grease, and cresols were not detected in the reagent mixture. The Soliditech reagent mix could not be reliably analyzed for phenol, p-cresol, and o-cresol because of its high alkalinity.

Leaching/extraction tests were performed using both destructive methods (TCLP, EP Toxicity, and BET) and non-destructive methods (ANS 16.1 and WILT). Destructive methods crush or grind the samples prior to testing destroying the physical integrity of solidified waste samples. Non-destructive methods were applied to intact cast cylinders.

Arsenic, lead, and zinc were found in EP, TCLP, and BET leachates from the pretreatment wastes. TCLP leachate from the pretreatment filter cake waste contained 4.3 mg/L of lead. These metals were not detected in post-treatment waste leachates produced by the EP, TCLP, and BET. The high alkalinity of the waste solidified by the Soliditech process neutralized the acidity of the EP and TCLP procedures. Pretreatment wastes could not be tested by ANS 16.1. Metals were not detected in post-treatment waste leachates produced by the ANS 16.1 method.

The post-treatment TCLP leachates contained phenol (100 to 630 µg/L), p-cresol (110 to 440 µg/L), and o-cresol (13 to 88 µg/L). These compounds were either not found or were found in lower concentrations in the pre-treatment waste samples.

Oil and grease were generally detected at similar concentrations (ND to 12 mg/L) in the EP, TCLP and BET leachates of both pretreatment and post-treatment wastes.

Results from the first 16 weeks of leaching by the Waste Interface Leaching Test (WILT) indicate that potentially toxic metals and organic compounds released by the solidified wastes were below levels of concern.

2.0 INTRODUCTION

This section provides background information about the Superfund Innovative Technology Evaluation (SITE) program, the Soliditech, Inc. solidification/stabilization technology, and the location of the demonstration. It also presents the demonstration program objectives, the technology evaluation criteria, a description of the field operations, and the project organization.

2.1 BACKGROUND

Past hazardous waste disposal practices and the environmental and human health impacts of those practices caused Congress to enact the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980. The original act established a Hazardous Substance Response Trust Fund to deal with costs incurred in cleanups of hazardous materials; this fund has become known as the Superfund. U.S. EPA has proceeded to investigate potentially dangerous hazardous waste sites and to establish national priorities for site cleanups. The ultimate objective of these investigations is to develop plans for permanent, long-term site cleanups. The list of potential cleanup sites is known as the National Priorities List (NPL).

Congress expressed concern over the use of land-based disposal and containment technologies to mitigate problems caused by releases of hazardous substances at hazardous waste sites. In response to this concern, the 1986 reauthorization of CERCLA, called the Superfund Amendments and Reauthorization Act of 1986 (SARA), mandates that, to the maximum extent practicable, U.S. EPA select remedial actions at Superfund sites that create permanent solutions to effects on human health or the environment. In doing so, U.S. EPA is directed to make use of innovative, alternative, or resource recovery technologies.

2.1.1 SITE Program

U.S. EPA has established the SITE program to accelerate the development, demonstration, and use of new or innovative technologies that offer permanent site cleanup. The program is administered by the Office of Solid Waste and Emergency Response (OSWER) and the Office of Research and Development (ORD).

Each year U.S. EPA solicits proposals to demonstrate innovative technologies. The most promising technologies are chosen for participation in the SITE demonstration program. OSWER, ORD and U.S. EPA regional personnel match these technologies with a list of potentially appropriate sites.

The demonstration program is designed to develop detailed and reliable performance and cost data on the innovative alternative technologies, so that potential users have sufficient information to make sound judgments as to the applicability of the technology to a specific site and to compare it to other currently available technology alternatives.

The program also identifies the governmental policy and regulatory requirements applicable to the technology and the hazardous substances being treated or destroyed.

2.1.2 Soliditech Technology

The Soliditech, Inc. solidification/stabilization technology mixes a proprietary reagent, called Urrichem, with a pozzolanic materials (such as fly ash), or cement, and other proprietary additives to solidify solids and sludges containing the organic and inorganic chemicals found at uncontrolled hazardous waste sites (Soliditech, 1987). The Soliditech process claims to chemically and physically immobilize hazardous constituents in waste material. This immobilization occurs by one or more of the following processes: encapsulation, adsorption, and incorporation into the crystalline structure of the solidified material. The final product is claimed to be a monolithic material with measurable structural strength and significantly reduced leaching potential.

The Soliditech technology was developed in the early 1980s and has been used mainly to treat industrial sludge. Typical Soliditech projects to date include the bench-scale testing and treatment of liquid, semi-liquid, and solid waste materials.

A more detailed process description is provided in Section 4.

2.1.3 Site Background

The site selected for the Soliditech, Inc., demonstration was the Imperial Oil Company, Inc./Champion Chemical Company site in Morganville, New Jersey. The location of the site is shown in Figure 1. This site is on the National Priority List (NPL) of Superfund clean-up sites. In the past, facilities located on the site processed tomatoes, produced various chemicals, and recovered waste oil. Present facilities blend and package oils for sale. The site includes a waste pile containing oil-saturated waste filter cake material, an abandoned storage tank containing oily

FIGURE 1

SITE LOCATION MAP

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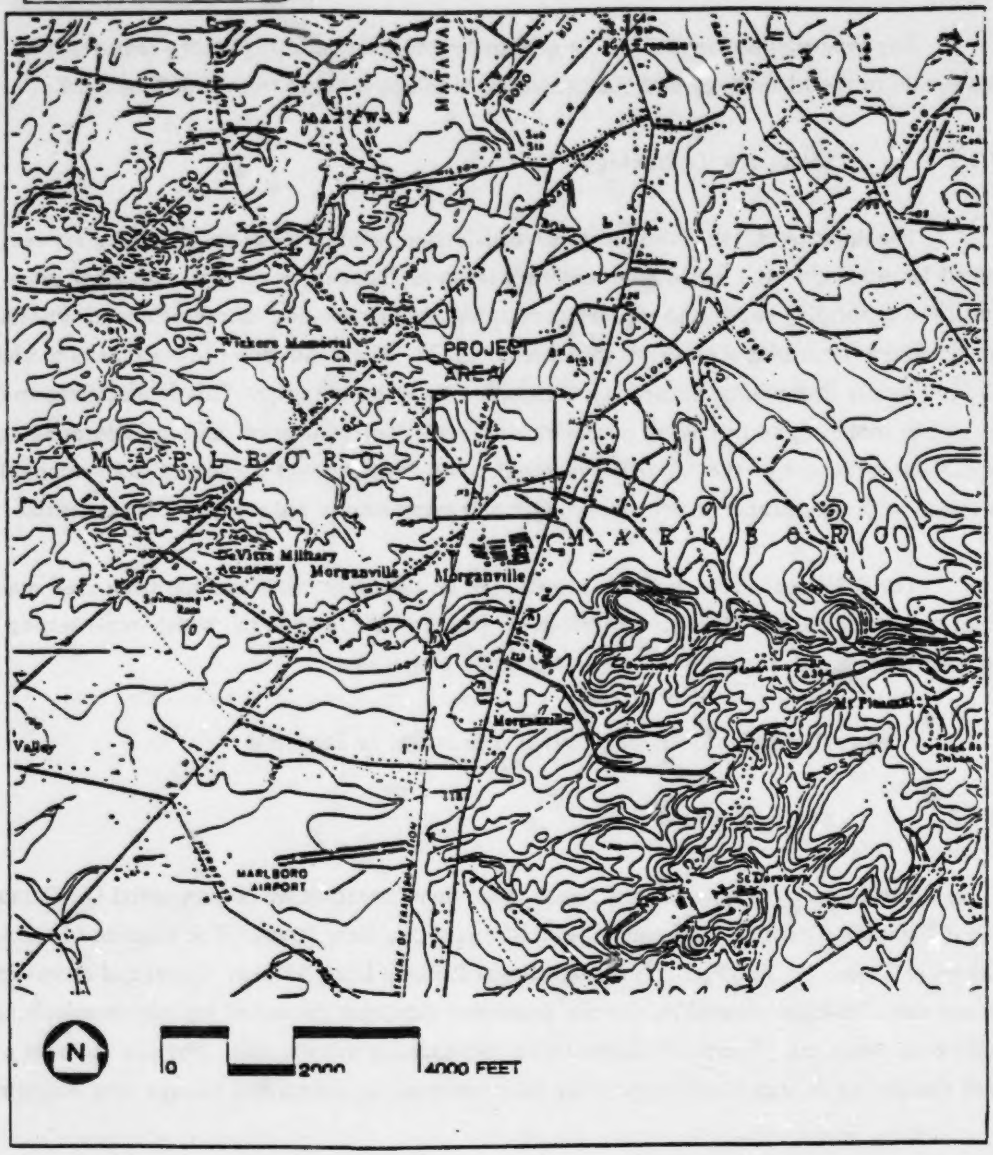
best available copy.



Source: E. C. Jordan Co., 1987.

sludge, several locations where oily sludge and/or waste filter cake material appear to have either been deposited or to have migrated, and other areas of surface and subsurface contamination. The locations of these areas are shown in Figures 2 and 3. (Although Off-Site Areas One and Two are referred to as "off-site," they are actually within the boundaries of the Superfund-designated site.)

Based upon past studies, including a treatability and site screening study, chemicals of concern at this site include petroleum hydrocarbons, other organic chemicals including PCBs, and metals (E.C. Jordan Co., 1987 and Radian, 1988).

2.2 TECHNOLOGY DEMONSTRATION PROGRAM OBJECTIVES

The SITE program mandate is to seek cost-effective alternatives to the traditional practice of using land disposal and containment for the remediation of hazardous waste sites. To address this mandate, the following objectives were developed for the Soliditech SITE demonstration:

- Determine the effectiveness of the technology to solidify and stabilize waste materials found at the site.
- Determine whether the solidified material maintains its structural properties and stabilization effectiveness over a five-year period.
- Determine the volume and mass change of the waste material after treatment with the pozzolan, reagent, and additive mix.
- Develop reliable capital and operating costs for the technology that can be used in the Superfund decision-making process.

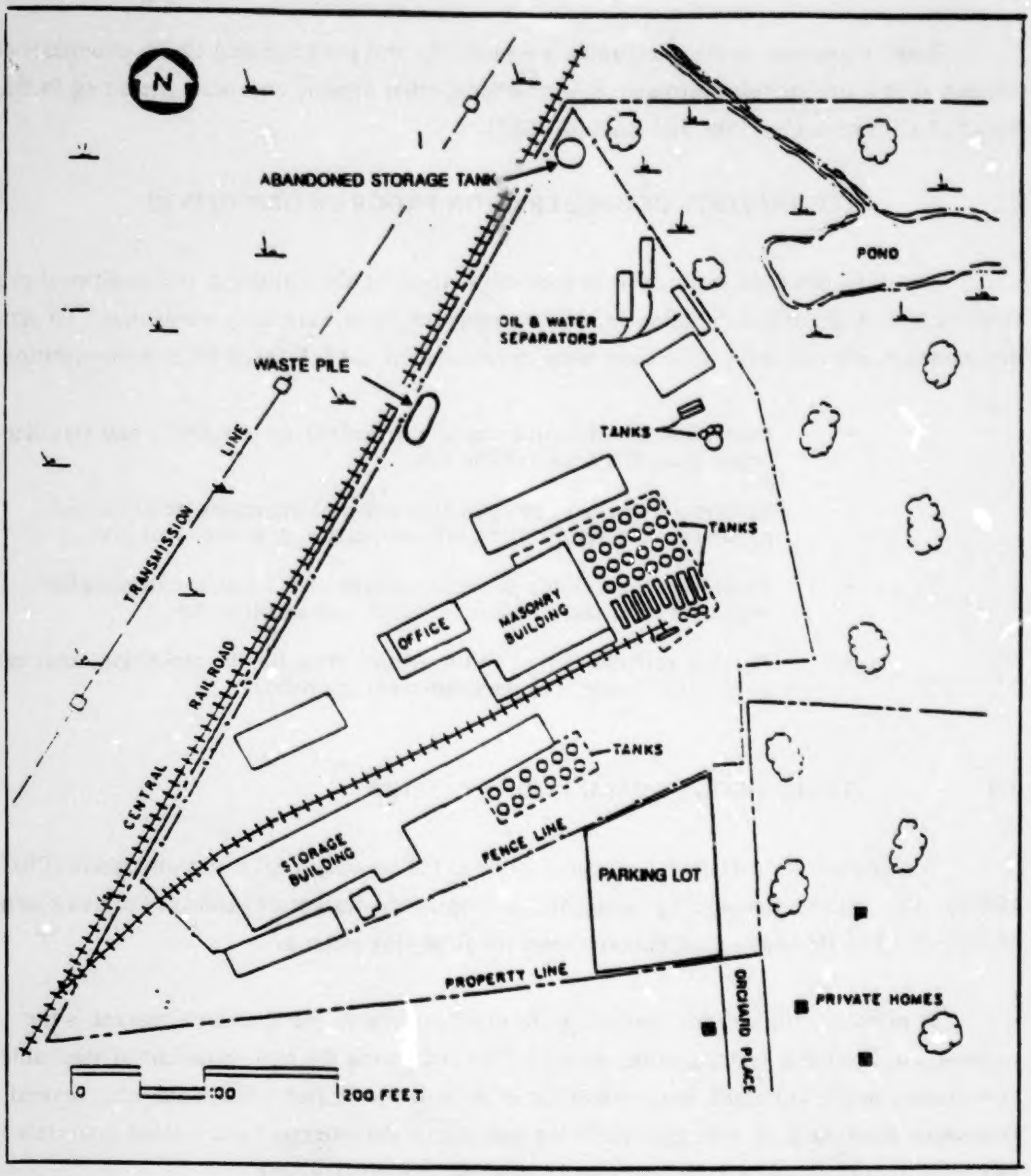
2.3 TECHNOLOGY EVALUATION CRITERIA

A Demonstration Plan was prepared prior to the Soliditech SITE demonstration (PRC, 1988b). This plan contained a Sampling and Analysis Plan designed to address the demonstration objectives. The Demonstration Plan outlined the following criteria.

A primary criterion for evaluating the effectiveness of the Soliditech process is the reduction of leachable contaminants, measured by comparing the concentrations of leachable constituents in the untreated waste materials to those in the treated waste materials. Several laboratory leaching tests were applied to the analysis of the untreated and treated materials. Each test attempted to simulate a different set of environmental conditions.

FIGURE 2

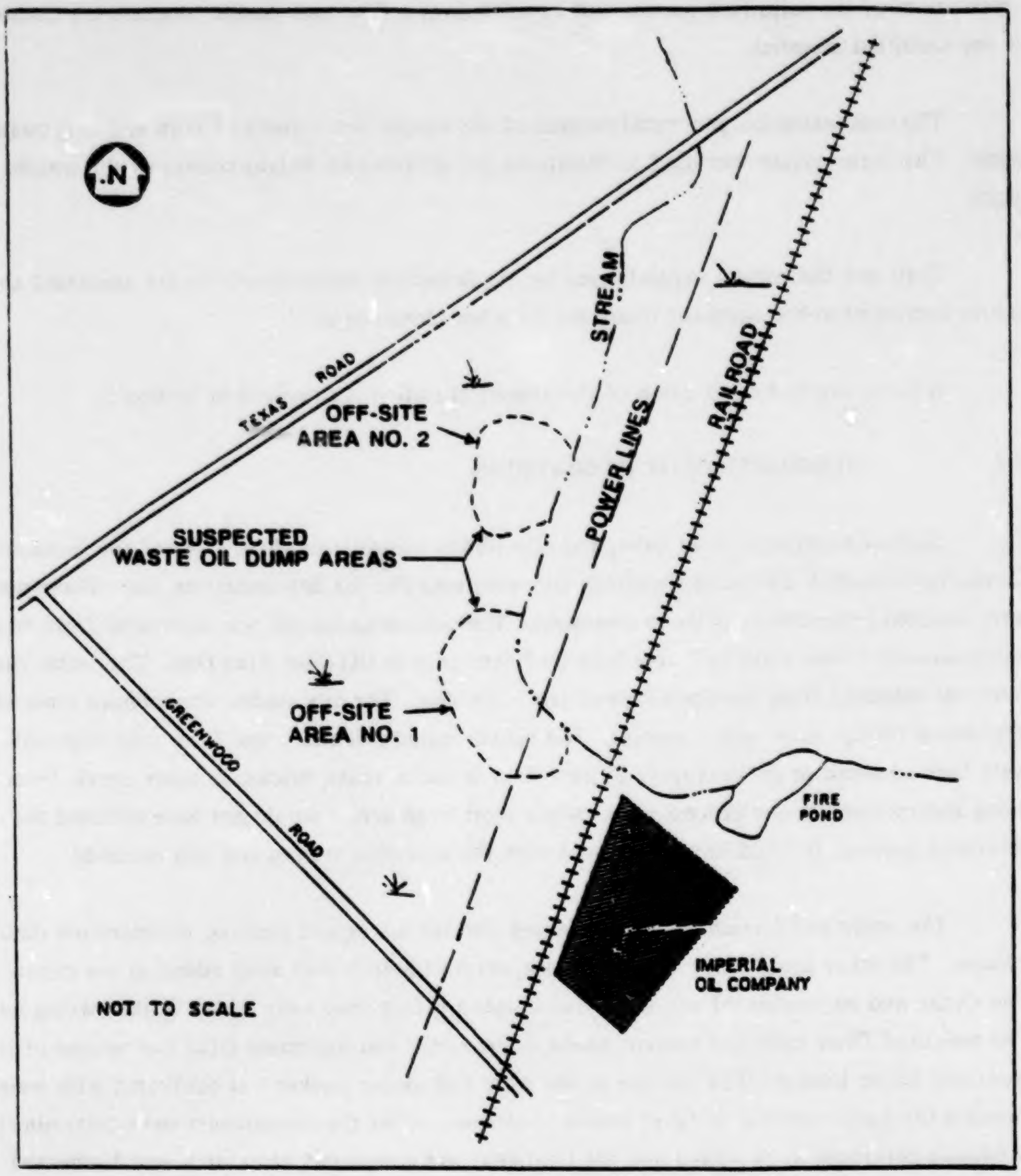
IMPERIAL OIL COMPANY/CHAMPION CHEMICALS SITE WASTE PILE AND ABANDONED STORAGE TANK



Source: Modified from E.C. Jordan, 1987.

FIGURE 3

IMPERIAL OIL COMPANY/CHAMPION CHEMICALS SITE OFF-SITE AREAS



Source: Modified from E.C. Jordan, 1987.

Another criterion for determining the effectiveness of the Soliditech process is the physical properties of the solidified material, such as unconfined compressive strength, permeability, microstructural changes, wet/dry durability, and freeze/thaw durability. These tests produce information about the long-term potential for water to permeate through a stabilized waste and about the structural integrity of a stabilized solid. Leaching tests and field observations of the solidified masses will be made over a five-year period to assess the durability of the solidified material.

The demonstration generated records of the weight and volume of both raw and treated waste. This information was used to determine the volume and weight change of the treated waste.

Costs and the capital expenditures for the Soliditech demonstration were generated and can be compared to the costs for treatment by other technologies.

A more detailed description of the analytical testing is provided in Section 5.

2.4 DESCRIPTION OF OPERATIONS

Contaminated soil, filter cake, and oily sludge wastes from three areas of the Imperial Oil Company/Champion Chemical Company site were used for the demonstration test. These wastes were sampled immediately prior to treatment. The contaminated soil was excavated from an area approximately 5 feet wide by 8 feet long by 3 feet deep at Off-Site Area One. The waste filter cake was collected from the open face of the waste pile. The oily sludge was scooped from the abandoned storage tank with a bucket. The contaminated soil and waste filter cake materials were both screened to prevent large objects such as rocks, roots, bricks, or other debris from being incorporated in the process runs. While most large debris would not have affected the Soliditech process, it could have interfered with the analytical testing and was removed.

The water and Urrichem, were pumped into the mixer and metered to determine their volume. The other proprietary additives were weighed before they were added to the mixer. The sludge and contaminated soil were also weighed before they were added to the mixing unit. The weight of filter cake and cement added to the mixer was estimated from the volume of the front end loader bucket. The volume of the front end loader bucket was calibrated with weighed amounts (55-gallon drums) of filter cake and cement. After the components were determined by Soliditech personnel to be mixed and the treatment was completed, the slurry was discharged from the mixer into one-cubic-yard plywood forms. Small waxed cardboard and PVC cylindrical

forms were filled from the slurry in the large plywood forms. After curing and transport to the analytical laboratory, these small forms were removed and the resulting cylindrical samples were used for analytical testing.

2.5 PROJECT ORGANIZATION

For the SITE program demonstration a cooperative agreement was signed between U.S. EPA and Soliditech, Inc. Soliditech was responsible for operating its equipment and providing the chemical additives. U.S. EPA, through its contractor, prepared the Demonstration Plan, performed the test site preparation, performed the sampling and analyses, evaluated the data, and prepared the Technology Evaluation Report. The field operations at the Imperial Oil Company/Champion Chemical Company site were performed by a U.S. EPA contractor.

3.0 CONCLUSIONS

Approximately 350 samples were collected during the Soliditech SITE demonstration. The data generated from the analyses of these samples as well as cost data were used to evaluate the Soliditech technology. The evaluation procedures used are standardized analytical methods prepared by the U.S. Environmental Protection Agency (U.S. EPA), American Society of Testing and Materials, and American Society of Agronomy. The analytical methods, as prescribed in the Demonstration Plan (PRC, 1988b), were peer-reviewed and approved by U.S. EPA.

3.1 SUMMARY OF PERFORMANCE DATA

Three waste materials -- contaminated soil, filter cake, and a mixture of filter cake and oily sludge -- were treated by the Soliditech solidification/stabilization process. These materials contained metals, volatile and semivolatile organic compounds, and PCBs.

Waste samples were analyzed prior to and after treatment to determine their physical and chemical characteristics. These data are reported in Sections 7.1 -- Physical Tests, and 7.2 -- Chemical Tests. The reagent mix used in the Soliditech process was analyzed to ensure that contaminants were not added to the wastes. Post-treatment samples were analyzed to determine the extent of stabilization of the wastes, in terms of physical and chemical characteristics. These data are reported in Sections 7.3 -- Leaching Tests, and 7.4 -- Morphological Tests. These analyses consisted of both total waste analyses and chemical analyses of leachates generated from both the untreated and treated wastes. Leaching/extraction tests were performed using both destructive methods (TCLP, EP Toxicity, and BET) and non-destructive methods (ANS 16.1 and WILT). Destructive methods crush or grind the samples prior to testing destroying the physical integrity of the solidified waste samples. Non-destructive methods were applied to intact cast cylinders. In addition, long-term testing is being conducted to evaluate the effectiveness of the solidification/stabilization technology over a period of five years. This section presents a summary of the results.

1. For the pretreatment filter cake/oily sludge samples, concentrations of volatile organic compounds (VOCs) ranged from 1.6 to 32 mg/Kg. For the pretreatment Off-Site Area One samples, concentrations of VOCs ranged from 2.2 to 8.3 mg/Kg. VOCs were not detected in the pretreatment filter cake samples or in the post-treatment waste samples.

2. Semivolatile organic compound (SVOC) analyses detected o-cresol in one of three replicate pretreatment filter cake/oily sludge samples. Post-treatment samples of the

filter cake/oily sludge and filter cake contained phenol (4.8 and 12 mg/Kg), p-cresol (4.4 and 14 mg/Kg), and 2,4-dimethylphenol (3.7 mg/Kg and ND). 2-Methylnaphthalene was detected in the filter cake/oily sludge and Off-Site Area One samples (4.4 and 3.8 mg/Kg). SVOCs were not detected in the reagent mix samples.

3. Metals were detected in pretreatment, reagent mix, and post-treatment samples. Levels of metals in the post-treatment waste were generally lower than those detected in the pretreatment waste.

4. Aroclor 1242 and Aroclor 1260 (PCBs) were detected in both the pretreatment and post-treatment wastes at levels ranging from 28 to 43 and 15 to 40 mg/Kg, respectively. PCBs were not detected in the reagent mix samples.

5. The pH of the pretreatment Off-Site Area One waste was 7.9. The filter cake and the filter cake/oily sludge wastes both had a pH of about 3.5. The post-treatment wastes and the reagent mix samples had pH values of about 12, which was attributed to the alkalinity associated with the cement in the solidification/stabilization mix.

6. The Eh (oxidation/reduction potential) of a water extract of the pretreatment wastes ranged from 100 millivolts (mv) to 370 mv. The Eh of a water extract at the post-treatment waste and reagent mix samples ranged from -31 to -63 mv.

7. The loss on ignition (a measure of water and organic content lost through volatilization) for the pretreatment waste samples ranged from 36 percent to 70 percent, and from 34 percent to 41 percent for the post-treatment waste samples.

8. The oil and grease content of the pretreatment waste samples ranged from about 2.8 percent to 17 percent. The post-treatment samples ranged from 4.6 percent to 7.7 percent.

9. TCLP Extraction Tests - PCBs were not detected in TCLP extracts of the pretreatment or post-treatment samples. Oil and grease was detected in both the pre- and post-treatment samples at concentrations of 1.4 to 12 μg/L. Lead and arsenic were found at levels as high as 5.4 mg/L and 0.19 mg/L, respectively, in the pretreatment samples. These metals were either not detected or were detected at lower levels in the post-treatment samples (see Tables 3 through 5). Barium, calcium, and sodium levels increased in the post-treatment samples due to the cement and proprietary additives used during treatment.

VOCs were detected at low levels in the reagent mix samples but were not detected in the post-treatment samples. Toluene (55 and 270 µg/L) and total xylenes (57 and 26 µg/L) were found in the pretreatment filter cake/oily sludge and Off-Site Area One TCLP leachates. Acetone was detected in all pretreatment wastes, but was also present in laboratory control blanks. SVOCs were not detected in the reagent mix samples (only post-treatment samples analyzed) or the filter cake pretreatment samples. In the pretreatment filter cake/oily sludge and Off-Site Area One samples, SVOCs (o-cresol, p-cresol, 2,4-dimethylphenol, phenol, and benzyl alcohol) were present at concentrations of 10 to 200 µg/L. The post-treatment samples from all areas contained higher levels of these compounds, ranging from 13 to 630 µg/L. The sources of these compounds are not known. The pH ranged from 4.6 to 5.1 for the pretreatment samples and from 10.8 to 11.6 for the post-treatment samples. These results indicate that alkalinity added by the Soliditech process greatly exceeded the acidic capacity of the TCLP. Table 17 in Section 7 present these results.

10. EP Toxicity Tests - PCBs were not detected in EP extracts of the pre- or post-treatment samples. Oil and grease was not found in the pretreatment samples except for the Off-Site Area One at 2.6 mg/L. Lead and zinc were detected at levels less than 1 mg/L in the pretreatment samples. These metals were not detected in the post-treatment samples. Barium, calcium, and sodium levels increased in the post-treatment samples. The pH ranged from 3.8 to 4.8 for the pretreatment samples and from 10.9 to 11.8 for the post-treatment samples. Table 18 in Section 7 present these results.

11. BET Extraction Tests - PCBs were not detected in BET extracts of the pretreatment or post-treatment samples. The pH for the filter cake and filter cake/oily sludge pretreatment samples ranged from 3.5 to 4.4; the pH for Off-Site Area One ranged from 8.3 to 9.0. Oil and grease ranged from less than 0.4 mg/L to 16 mg/L for the pretreatment BET samples. Post-treatment oil and grease levels ranged from less than 0.4 mg/L to 26 mg/L. The concentration of metals detected in the pretreatment and post-treatment BET samples increased as the solid to liquid ratio of the extract decreased. Lead and zinc were not detected in any of the post-treatment samples. Tables 19 to 22 in Section 7 present these results.

12. ANS 16.1 Leaching Tests - This non-destructive leaching test could only be performed on treated waste samples. PCBs were not detected in the leachates. Oil and grease was not detected in the filter cake and filter cake/oily sludge samples, but was detected in the Off-Site Area One sample at 1.1 mg/L to 3.2 mg/L. Lead and zinc were found at the

detection limits in some of the samples, and arsenic was detected at 0.0053 to 0.0080 mg/L in the Off-Site Area One samples. The pH of leachates ranged from 10.7 to 11.7. Tables 23 through 25 in Section 7 present these results.

13. **Morphological features will be determined through radiographic X-ray diffraction and scanning electron microscope (SEM) analyses. There are no results to report at this time.**

14. **Physical Tests - The bulk density of the pretreatment wastes ranged from 1.14 to 1.26 g/cm^3. The bulk density of the post-treatment wastes ranged from 1.43 to 1.68 g/cm^3. The moisture content in the pretreatment samples ranged from 23.5 to 58.1 percent, and from 12.6 to 21.0 percent in the post-treatment samples.**

The mean particle size for the pretreatment wastes ranged from 0.32 to 0.46 mm. Particle size on the post-treatment wastes was not determined because the wastes were monolithic solids.

Permeability of the pretreatment waste was not determined. Permeability of the post-treatment waste ranged from 8.9 x 10^{-9} cm/sec to 4.5 x 10^{-7} cm/sec for the filter cake/oily sludge and filter cake samples, respectively.

The unconfined compressive strengths ranged from 390 psi for treated filter cake to 860 psi for treated filter cake/oily sludge.

Results of the wet/dry weathering tests for all of the post-treatment wastes indicate no measurable weight loss from the 12 wet/dry cycles.

No weight loss greater than one percent occurred in the post-treatment cores as a result of the 12 freeze/thaw cycles.

The acid neutralization capacity of the pretreatment waste was low in all cases. However, adding the solidifying proprietary additives increased the alkalinity of the treated wastes to extremely high levels.

15. **Results from the first 16 weeks of leaching by the WILT procedure indicate that potentially toxic metals and organic compounds released from the solidified wastes were below levels of concern. Measurable concentrations of major cations and oil and grease**

were found in leachates. The pH of leachates ranged from 10.9 to 12.7. These preliminary data are presented in Appendix G.

Tables 1 and 2 summarize the physical and chemical properties of the untreated and treated wastes. Tables 3 through 8 summarize the percent changes in TCLP, EP, BET, and ANS 16.1 leachate concentrations due to solidification/stabilization for various analytes of concern. The percent/reduction (or increase) values have been corrected to account for the dilution of wastes by adding cement, water, Urrichem, and proprietary additives.

In summary, data from all extraction and leaching tests showed negligible release of contaminants. Neither PCBs nor volatile organic compounds were detected in the TCLP extracts of treated wastes. Significantly reduced amounts of metals were detected in the TCLP, EP, BET, and ANS 16.1 extracts of treated wastes compared to untreated wastes. However, portland cement contributed several metals to the treated waste. Low concentrations of phenols and cresols were detected in post-treatment TCLP extracts. These compounds were possibly formed during the stabilization reactions. The pH values of the treated wastes were near 12.

Physical stability of the treated wastes was high. Unconfined compressive strength of the treated waste was significant. Permeability values for the treated wastes were very low. Weight loss of the treated wastes after wet/dry and freeze/thaw cycles was negligible.

3.2 SUMMARY OF MAJOR PROBLEMS

No major operational problems were encountered during the Soliditech demonstration. Various minor problems that did occur are discussed in the appropriate sections of this Technology Evaluation Report.

3.3 ADVANTAGES AND LIMITATIONS OF THE SOLIDITECH PROCESS

The advantages and limitations of the solidification/stabilization process are summarized below.

3.3.1 Advantages of the Soliditech Process

- The equipment required for the process is relatively simple and easily transported on two flatbed truck trailers. A dry solids storage hopper and a mixer are the two major pieces of equipment. The minimal electrical power requirements for transfer of pozzolans from the hopper to the mixer can be met by a small,

TABLE 3

TCLP AND EP TOXICITY LEACHATE RESULTS FOR FILTER CAKE WASTE

	TCLP			EP Toxicity		
	Untreated	Treated	Percent Reduction (Increase)[a][b]	Untreated	Treated	Percent Reduction (Increase)[a][b]
PCBs (μg/L)						
Aroclor-1242	<0.42	<0.45	ND	<0.43	<0.41	ND
Aroclor-1260	<0.84	<0.90	ND	<0.86	<0.82	ND
Metals (AA) (mg/L)						
Arsenic	0.0050	<0.0020	ND	0.010	0.0023	61
Lead	NA	0.0020	ND	0.26	0.0023	99
Metals (ICPES) (mg/L)						
Barium	1.4	1.3	(57)	0.21	1.4	(1,000)
Cadmium	0.0052	<0.0050	ND	<0.0050	<0.0050	ND
Lead	4.3	<0.20	>92	0.25	<0.050	>66
Nickel	<0.020	<0.020	ND	<0.020	<0.020	ND
Zinc	0.28	<0.020	>88	0.032	<0.020	ND
Other Chemical Analyses						
Filterable Residue (TDS) (mg/L)	4,500	8,500	(220)	90	9,500	(18,000)
Oil & Grease, infrared (mg/L)	1.4	4.4	(430)	<0.40	4.0	(>1,600)

Notes

[a] Percent reduction (increase) was calculated after correcting for dilution due to treatment. The treated filter cake waste was 59 percent waste and 41 percent additives by weight.

Equation: $$\text{Percent Reduction (Increase)} = \frac{\text{Analyte Concentration Untreated} - \left(\frac{\text{Analyte Concentration Treated}}{\text{Dilution Ratio}}\right)}{\text{Analyte Concentration Untreated}} \times 100\%$$

[b] An increase in metal and TDS concentrations may be due to presence of metals and other inorganics in the reagent mixture.

NA = Not analyzed
ND = Not possible to determine
> = Greater than
< = Less than

TABLE 4

TCLP AND EP TOXICITY LEACHATE RESULTS FOR FILTER CAKE/OILY SLUDGE MIXTURE

	TCLP			EP Toxicity		
	Untreated	Treated	Percent Reduction (Increase)[a][b]	Untreated	Treated	Percent Reduction (Increase)[a][b]
PCBs (μg/L)						
Aroclor-1242	<0.43	<0.11	ND	<0.43	<0.42	ND
Aroclor-1260	<0.86	<0.21	ND	<0.86	<0.84	ND
Metals (AA) (mg/L)						
Arsenic	0.014	<0.0020	>64	0.011	0.0020	55
Lead	NA	0.014	ND	0.55	0.015	93
Metals (ICPES) (mg/L)						
Barium	2.5	5.1	(410)	1.1	5.7	(1,200)
Cadmium	0.0093	<0.0050	ND	0.0082	<0.0050	ND
Lead	5.4	<0.050	>98	0.52	<0.050	>76
Nickel	0.027	<0.020	ND	<0.020	<0.020	ND
Zinc	1.3	<0.020	>96	0.86	<0.020	>94
Other Chemical & Physical Tests						
Filterable Residue (TDS) (mg/L)	5,200	8,600	(310)	330	9,100	(6,800)
Oil & Grease, infrared (mg/L)	1.6	2.4	(270)	<0.40	3.1	(>18,000)

Notes

[a] Percent reduction (increase) was calculated after correcting for dilution due to treatment. The treated filter cake/oily sludge waste was 40 percent waste and 60 percent additives by weight.

$$\text{Equation: Percent Reduction (Increase)} = \frac{\text{Analyte Concentration Untreated} - \left(\frac{\text{Analyte Concentration Treated}}{\text{Dilution Ratio}}\right)}{\text{Analyte Concentration Untreated}} \times 100\%$$

[b] An increase in metal and TDS concentrations may be due to presence of metals and other inorganics in the reagent mixture.

NA = Not analyzed
ND = Not possible to determine
> = Greater than
< = Less than

TABLE 5

TCLP AND EP TOXICITY LEACHATE RESULTS FOR OFF-SITE AREA ONE WASTE

	TCLP			EP Toxicity		
	Untreated	Treated	Percent Reduction (Increase)[a][b]	Untreated	Treated	Percent Reduction (Increase)[a][b]
PCBs (μg/L)						
Aroclor-1242	<0.42	<0.44	ND	<0.45	<0.21	ND
Aroclor-1260	<0.84	<0.87	ND	<0.90	<0.42	ND
Metals (AA) (mg/L)						
Arsenic	0.19	0.017	85	0.18	0.028	73
Lead	0.55	0.012	96	0.12	0.012	83
Metals (ICPES) (mg/L)						
Barium	1.6	2.3	(150)	0.58	2.4	(610)
Cadmium	<0.0050	<0.0050	ND	0.0052	<0.0050	ND
Lead	0.46	<0.050	>81	0.067	<0.050	ND
Nickel	0.033	<0.020	ND	<0.020	<0.020	ND
Zinc	0.63	<0.020	>95	0.26	<0.020	>86
Other Chemical Analyses						
Filterable Residue (TDS) (mg/L)	6,300	9,000	(150)	790	9,400	(2,000)
Oil & Grease, infrared (mg/L)	1.9	12	(990)	2.6	11	(630)

Notes

[a] Percent reduction (increase) was calculated after correcting for dilution due to treatment. The treated Off-Site Area One waste was 58 percent waste and 42 percent additives by weight.

Equation:

$$\text{Percent Reduction (Increase)} = \frac{\text{Analyte Concentration Untreated} - \left(\dfrac{\text{Analyte Concentration Treated}}{\text{Dilution Ratio}}\right)}{\text{Analyte Concentration Untreated}} \times 100\%$$

[b] An increase in metal and TDS concentrations may be due to presence of metals and other inorganics in the reagent mixture.

ND = Not possible to determine
> = Greater than
< = Less than

TABLE 6

BET EXTRACT RESULTS FOR FILTER CAKE WASTE

	Solid to Liquid Ratios								
	1:4			1:20			1:100		
	Untreated	Treated	Percent Reduction (Increase)(a)(b)	Untreated	Treated	Percent Reduction (Increase)(a)(b)	Untreated	Treated	Percent Reduction (Increase)(a)(b)
PCBs (µg/L)									
Aroclor-1242	<0.42	<0.43	ND	<0.41	<0.41	ND	<0.42	<0.21	ND
Aroclor-1260	<0.84	<0.86	ND	<0.82	<0.82	ND	<0.84	<0.42	ND
Metals (AA) (mg/L)									
Arsenic	0.072	0.011	74	0.014	0.0037	55	0.020	0.0020	83
Metals (ICPES) (mg/L)									
Barium	0.14	6.3	(7,500)	0.28	3.4	(2,000)	0.47	0.92	(230)
Cadmium	0.0073	<0.0050	ND	<0.0050	<0.0050	ND	<0.0050	<0.0050	ND
Lead	0.87	<0.050	>90	0.42	<0.050	>80	0.18	<0.050	>53
Nickel	0.063	<0.020	>46	<0.020	<0.020	ND	<0.020	<0.020	ND
Zinc	0.27	<0.020	>87	0.047	<0.020	>28	0.020	<0.020	ND
Other Chemical Analyses									
Filterable Residue (TDS) (mg/L)	440	3,800	(1,400)	120	1,700	(2,300)	40	760	(3,100)
Oil & Grease, infrared (mg/L)	0.65	6.3	(1,500)	0.53	2.7	(770)	<0.40	<0.40	ND
Total Organic Carbon	91	140	(160)	28	43	(160)	11	14	(120)

Notes

a Percent reduction (increase) was calculated after correcting for dilution due to treatment. The treated filter cake waste was 59 percent waste and 41 percent additives by weight.

$$\text{Equation: Percent Reduction (Increase)} = \frac{\text{Analyte Concentration Untreated} - \left(\dfrac{\text{Analyte Concentration Treated}}{\text{Dilution Ratio}}\right)}{\text{Analyte Concentration Untreated}} \times 100\%$$

b An increase in metal and TDS concentrations may be due to presence of metals and other organics in the reagent mixture.

ND = Not possible to determine
> = Greater than
> = Less than

TABLE 7

BET EXTRACT FOR FILTER CAKE/OILY SLUDGE MIXTURE

	Solid to Liquid Ratios								
	1:4			1:20			1:100		
	Untreated	Treated	Percent Reduction (Increase)[(a)(b)]	Untreated	Treated	Percent Reduction (Increase)[(a)(b)]	Untreated	Treated	Percent Reduction (Increase)[(a)(b)]
PCBs (µg/L)									
Aroclor-1242	<1.1	<0.42	ND	<0.44	<0.42	ND	<0.41	<0.22	ND
Aroclor-1260	<2.2	<0.84	ND	<0.88	<0.84	ND	<0.82	<0.44	ND
Metals (AA) (mg/L)									
Arsenic	0.042	0.0080	52	0.035	0.0023	84	0.0083	0.0030	10
Metals (ICPES) (mg/L)									
Barium	0.83	17	(5,000)	0.78	9.6	(3,000)	0.48	2.6	(>1,300)
Cadmium	0.036	<0.0050	>65	0.0062	<0.0050	ND	<0.0050	<0.0050	ND
Lead	1.7	<0.050	>93	0.43	<0.050	>71	0.14	<0.050	>11
Nickel	0.049	0.023	(13)	0.028	<0.020	ND	0.022	<0.020	ND
Zinc	2.7	<0.020	>98	0.69	<0.020	>93	0.16	<0.020	>69
Other Chemical Analyses									
Filterable Residue (TDS) (mg/L)	1,800	3,500	(390)	470	2,300	(1,100)	110	1,200	(2,600)
Oil & Grease, infrared (mg/L)	3.2	4.9	(280)	2.2	1.3	(48)	1.3	0.43	17
Total Organic Carbon (mg/L)	200	110	(38)	60	32	(33)	21	8.0	5

Notes

a Percent reduction (increase) was calculated after correcting for dilution due to treatment. The treated filter cake/oily sludge waste was 40 percent waste and 60 percent additives by weight.

Equation: Percent Reduction (Increase) =

$$\frac{\text{Analyte Concentration Untreated} - \left(\frac{\text{Analyte Concentration Treated}}{\text{Dilution Ratio}}\right)}{\text{Analyte Concentration Untreated}} \times 100\%$$

b An increase in metal and TDS concentrations may be due to presence of metals and other organics in the reagent mixture.

ND = Not possible to determine
> = Greater than
> = Less than

TABLE 8

BET EXTRACT FOR OFF-SITE AREA ONE WASTE

	Solid to Liquid Ratios								
	1:4			1:20			1:100		
	Untreated	Treated	Percent Reduction (Increase)(a)(b)	Untreated	Treated	Percent Reduction (Increase)(a)(b)	Untreated	Treated	Percent Reduction (Increase)(a)(b)
PCBs (µg/L)									
Aroclor-1242	<1.1	<0.43	ND	<1.1	<0.21	ND	<0.43	<0.10	ND
Aroclor-1260	<2.3	<0.86	ND	<2.2	<0.42	ND	<0.86	<0.20	ND
Metals (AA) (mg/L)									
Arsenic	0.38	0.067	70	0.29	0.022	87	0.19	0.0097	91
Metals (ICPES) (mg/L)									
Barium	0.11	9.7	(15,000)	0.047	5.5	(20,000)	0.023	1.4	(10,000)
Cadmium	0.0068	<0.0050	ND	0.0055	<0.0050	ND	<0.0050	<0.0050	ND
Lead	<0.050	<0.050	ND	<0.050	0.090	(>210)	<0.050	<0.050	ND
Nickel	<0.020	<0.033	ND	<0.020	<0.020	ND	<0.020	<0.020	ND
Zinc	<0.020	<0.020	ND	<0.020	<0.020	ND	<0.020	<0.020	ND
Other Chemical & Physical Tests									
Filterable Residue (TDS) (mg/L)	1,100	4,600	(620)	390	2,600	(1,000)	330	980	(410)
Oil & Grease, infrared (mg/L)	16	26	(180)	12	15	(120)	4.4	3.7	(45)
Total Organic Carbon (mg/L)	190	120	(9)	73	54	(28)	30	14	20

Notes

a Percent reduction (increase) was calculated after correcting for dilution due to treatment. The treated filter cake/oily sludge waste was 40 percent waste and 60 percent additives by weight.

Equation: Percent Reduction (Increase) =

$$\frac{\text{Analyte Concentration Untreated} - \left(\dfrac{\text{Analyte Concentration Treated}}{\text{Dilution Ratio}}\right)}{\text{Analyte Concentration Untreated}} \times 100\%$$

b An increase in metal and TDS concentrations may be due to presence of metals and other organics in the reagent mixture.

ND = Not possible to determine
> = Greater than
> = Less than

transportable generator. The mixer is self-powered by a diesel engine. During the demonstration, the equipment appeared to be problem-free.

- The reagent and proprietary additives required for waste treatment are either readily obtainable, such as cement or pozzolans, and water; or are required in relatively small amounts that can be readily transported to the treatment location, such as Urrichem and the other proprietary additives.

- The conditions imposed upon Soliditech during the demonstration did not allow optimum processing of waste, because each test run treated a different type of waste. Nevertheless, it was apparent that the process was relatively easy to run and moderately fast. Approximately 5 to 10 cubic yards of waste can be treated in an hour, once the equipment is set up and all reagent, proprietary additives, and waste materials are ready to be added to the mixer.

- The Soliditech process is able to solidify both solid and semi-solid materials. The oily sludge found at the site was solidified after mixing it with some filter cake material from the waste pile. Solids such as rocks or other debris up to 4 inches in diameter can be accommodated by the process. The size of rocks and debris was limited in this demonstration because of test sample requirements, but techniques should accommodate any size particles and be limited only by the size of the equipment used.

3.3.2 Limitations of the Soliditech Process

- The process should only be used when the ambient temperature is above freezing or when the treated material can be maintained at above freezing temperatures during the first 24 hours after treatment. At lower temperatures the treated material may not adequately solidify. The temperature during the demonstration was above 35 degrees F during the day but below freezing at night. As a result, all analytical samples and one block of solidified material from each test run were allowed to cure in a heated warehouse at temperatures ranging from 50 to 70 degrees F.

- The process is generally limited to treating wastes with a pH of 2 to 12. Waste material with a neutral pH is ideal for treatment. The pH of the raw waste material in this case ranged from 3.4 to 7.9.

- Accurately determining of the weights of materials added to the mixer was required for the demonstration but was found to be difficult. If this information is necessary, more complicated gauges and weight feeders could be added to the process; however, this would increase the system's complexity.

- Since the Soliditech process is a batch process, a number of batches must be run to treat a large area. During the demonstration, 14 cubic yards of material were treated in four batches.

- The process has certain limits to the amount of water or oil and grease that can be accommodated without pretreatment of the waste. When large concentrations of these materials are present, adjustments to the amounts of proprietary additives must be made. Process limits on water and oil and grease content in the wastes must be determined on a case by case basis. Waste material treated during the demonstration contained up to 58 percent water, and 17 percent oil and grease and required no pretreatment.

- It is difficult to assess when the treated material is adequately mixed. Some minor problems were observed during the demonstration. The initial batch of treated waste material (filter cake/oily sludge) was not totally blended as demonstrated by unmixed lumps of waste material in the solidified product.

- The suitability of the process for treating waste containing volatile organic compounds is difficult to quantify as these chemicals may be lost during waste collection and treatment.

- The long-term stability of the treated waste material is not known. U.S. EPA will monitor the solidified wastes for the next five years.

4.0 SOLIDITECH PROCESS

4.1 PROCESS DESCRIPTION

The Soliditech, Inc. solidification/stabilization process uses a batch process to treat waste material. A schematic diagram of this process is shown in Figure 4. The operating capacity of the process is governed by the size of the mixer, the amount of time required to load and discharge the mixer, and the amount of mixing time required to achieve homogeneous mixing of the waste material and the reagent and additives. The two mixers used during the demonstration had nominal capacities of 2- and 10-cubic yards.

The following materials were added to the Soliditech mixing unit during the demonstration (Soliditech, 1989a), measured either by weight or volume.

- Waste Material (contaminated soil and sludge by weight and filter cake by volume)
- Water (volume)
- Urrichem (volume)
- Proprietary Additives (weight)
- Pozzolanic Material/Cement (volume)

Based on treatability studies of samples of waste material collected at the Imperial Oil site (PRC, 1988a), Soliditech optimized treatment formulations for each of the waste materials to be treated. The formulations are presented in Table 9 (Soliditech, 1989a). As this table shows, the amounts of the reagent, proprietary additives, pozzolanic material, and water were approximately proportional to the amount of waste treated.

4.2 PROCESS SCHEMATIC

The Soliditech process, as shown in Figure 4, includes the following operations:

- A measured amount of waste material is added to the mixer through the screen welded to the mixer top. This is accomplished by a front-end loader or drum grappler.
- Measured amounts of water, reagent, and proprietary additives are added to the mixer by pump, front-end loader, or drum grappler and thoroughly mixed with the waste.

TABLE 9

SOLIDITECH TREATMENT FORMULATIONS

	Estimated Weights of Materials (lbs.)[a]			
	Reagent Mixture	Filter Cake/ Oily Sludge	Filter Cake	Off-Site Area
Waste Material	800[b]	3,950	11,200	9,100
Type II Cement[c]	442	4,970	4,920	4,540
Urrichem	8.96	39.2	111	90.7
Additives	16.5	140	167	136
Water	154	666	2,687	1,830
TOTAL WEIGHT	1,420	9,760	19,100	15,700

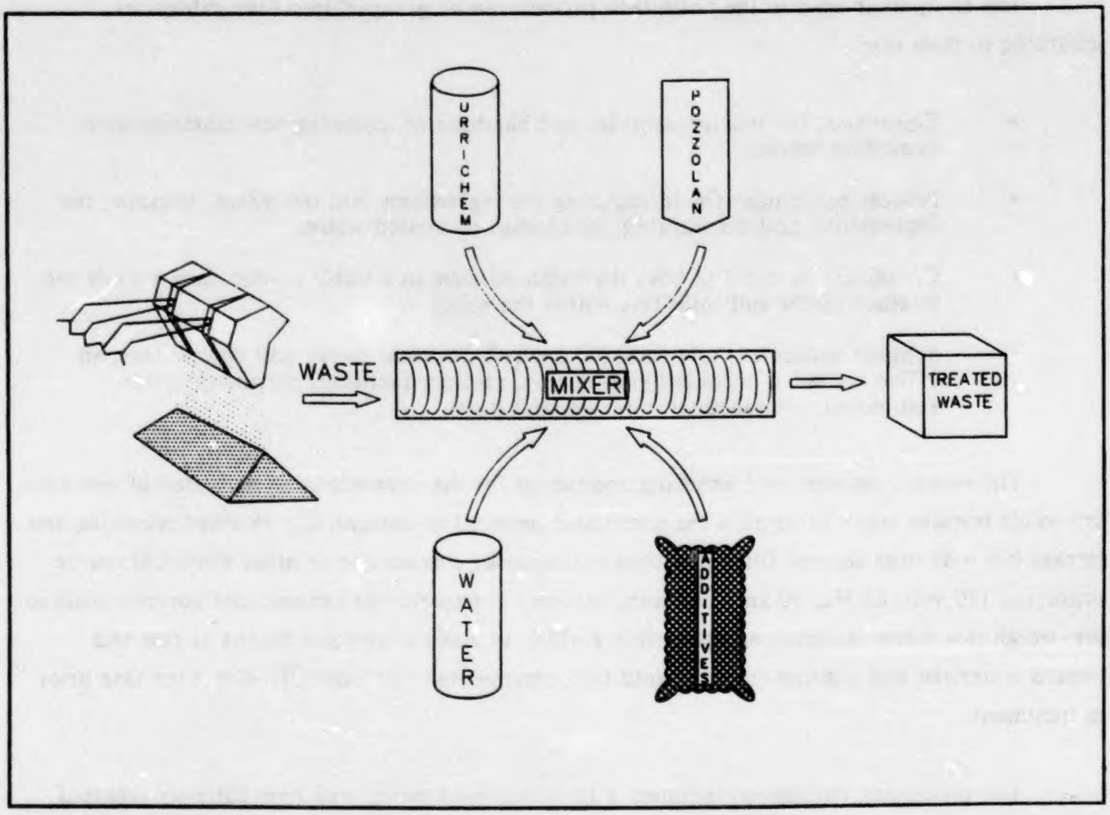
Notes:

[a] The weights for cement, filter cake, Urrichem and water were calculated based on volume.

[b] Clean sand used rather than waste material for this test run.

[c] According to Soliditech, fluffing of the cement may cause the cement weight values to be as much as 5 percent higher than the actual weight, because the weight of cement was estimated from volume added.

FIGURE 4
SOLIDITECH PROCESS SCHEMATIC



Source: Soliditech, 1987.

- The cement is then measured and added to the mixer using the front-end loader.
- The resulting mixture is mixed for approximately 40 to 60 minutes.
- The treated waste mixture is discharged to one-cubic yard forms. Samples to be used for analyses are collected directly from the cubic-yard forms. All treated material is allowed to cure for 28 days. At this time, the solidified treated waste material is removed from the forms and sample containers.

4.3 EQUIPMENT SPECIFICATIONS

The equipment used in the Soliditech process can be grouped into four categories, according to their use:

- Equipment for receipt, storage, and handling of incoming raw materials and hazardous wastes.
- Process equipment for introducing the ingredients into the mixer, blending the ingredients, and discharging the batches of treated waste.
- Containers or mold to hold the batch mixture in a stable configuration while the mixture reacts and solidifies within the mold.
- Support equipment and facilities such as electrical power and power lines, an office trailer, a decontamination area, sanitary facilities, decontamination equipment, maintenance supplies, and tools.

The receipt, storage, and handling equipment for the demonstration consisted of one bulk, dry solids transfer truck to contain the pozzolanic material or cement; one elevated receiving and storage bin with dust control filter and blower (requiring a generator or other electrical source supplying 120 volt, 60 Hz, 30 amp electrical service) to transfer the cement; one portable scale to pre-weigh raw waste material; an all-terrain forklift to move drums and blocks of raw and treated materials; and a dump truck to hold the contaminated soil from Off-Site Area One prior to treatment.

The processing equipment included a 10-cubic-yard mixer with hydraulically actuated legs, a hydraulic agitator, a screen with 4-inch by 4-inch openings to trap and remove any large roots, debris, or other material from the waste to be treated, and a small 2-cubic-yard mixer. The large Soliditech mixer with attached screen is pictured in Figure 5. The small mixer was brought to the site to demonstrate that small volumes of waste could also be treated by the Soliditech process. This mixer was used to process sand, Urrichem, cement and proprietary additives for the reagent mix test run.

The molds were reinforced 1-cubic-yard plywood forms. Treated waste material from the mixing unit was discharged into these forms. An all-terrain forklift was used to move and store the filled forms.

Equipment was decontaminated with a high-pressure steam cleaner. Waste water and solid residual material from decontamination was collected and stored for disposal by New Jersey Department of Environmental Protection (NJ DEP).

The following specifications describe the equipment in more detail, including capacities, dimensions, functions, and optional equipment:

- Pozzolanic Material/Cement Bin and Hopper -- This upright aluminum hopper can discharge through a flexible hose. The hopper is 9- by 9- by 16-feet high and feeds down to an 8-foot-high cone bottom. The hopper is supported on four, 10-foot-legs. During the demonstration the cement was transferred from the hopper through the hose to the bucket of a front-end loader that transferred the cement to the mixer. The bucket of the front-end loader was previously calibrated. Dust emissions during filling and removal were controlled by a baghouse.

- Bulk Cement Storage Trailer -- The bulk transport trailer that delivered the cement for the demonstration had a hopper adequate to hold 12 tons of cement. The cement was transferred from the hopper of this trailer to the storage bin and hopper. Dust emissions during filling and removal were controlled by a baghouse.

- Reagent Storage Tank and Metering System -- The reagent storage system consisted of two 350-gallon steel tanks mounted on the mixer transport trailer, and a metering pump capable of displacing an accurate volume of liquid. The pump was used to transfer reagent from the storage tank to the mixing unit.

- Mixing Unit -- The large Soliditech mixer is a narrow, low profile, totally independent and self-contained, 10-cubic-yard unit measuring 20 feet long, 8 feet wide, and 4 feet high. It is mounted on a low-boy trailer for total mobility. The mixing unit has a variable speed agitator and specialized lifting mechanism that allows for total mixing and complete discharge of mixed product. The open top allows for visual inspection of consistency and volume as well as easy loading from front-end loaders, and drums. For the demonstration, a screen with 4-inch by 4-inch openings was welded to the top of the mixer and used for screening waste material. The mixing unit has an independent four-point body hoist that can raise the mixing unit to a height of 6 feet, thus allowing the mixing unit to be placed above drums, plywood forms, or a feed hopper for easy discharge of the treated waste material with minimal handling and contact.

  The mixer blade consists of a full sweep, bi-directional paddle mixer with mixing speeds between 2 and 16 revolutions per minute. Each paddle sweeps the bottom of the shell for complete mixing, discharge, and clean-out of the materials, with no dead areas of mixing. The mixing unit contains an eccentric, weighted, hydraulic vibrator rated at 2,000 revolutions per minute to aid clean out. All mixer controls are located at the rear of the mixing unit.

The small Soliditech mixer is a self-contained, 2-cubic yard unit measuring 6 feet long, 3 feet wide, and 5 feet high. It is mounted on a trailer. It is operationally similar to the large Soliditech mixer.

5.0 DEMONSTRATION PROCEDURES

This section presents the site description, the waste characteristics, the sampling and analysis plan, and the QA/QC plan for the Soliditech demonstration.

5.1 SITE DESCRIPTION

The Soliditech technology demonstration was conducted at the Imperial Oil Company/Champion Chemical Company site in Morganville, Monmouth County, New Jersey. The site is located on Orchard Place in Morganville, approximately 1/2 mile northwest of the junction of Rt. 3 and Rt. 79 (Figure 1). The site is situated in a largely rural area with scattered residential properties along surrounding roads. A commercial shopping center is located 1/2 mile southeast of the site, and two automobile scrap yards are located just northwest of the site. Lake Lefferts, a swimming and recreational area, is located approximately 1 mile north of the site. Lake Lefferts receives surface-water drainage from the watershed that contains the Imperial Oil site. The average annual temperature (Newark) is approximately 52° F and the monthly average temperatures range from approximately 32° F in January and February to 76° F in July.

The site is divided into the area presently occupied by the Imperial Oil Company facility (On-Site Area) and certain immediately surrounding areas (Off-Site Areas One and Two). These two areas of the Superfund site are shown in Figures 2 and 3. For the purposes of this SITE demonstration, the NPL definition of the Imperial Oil site will be used. The property occupied by the Imperial Oil Company, where the demonstration was performed, is enclosed by a security fence. The surrounding areas, including the areas referred to as Off-Site Area One and Off-Site Area Two, are not fenced. The off-site areas are undeveloped.

The on-site area immediately adjacent to the waste pile is underlain by 2 to 10 feet of fill material, consisting of sand, silt, and gravel and containing varying amounts of ash, oil sludge, waste filter cake, wood fragments, coal, bricks, concrete rubble, vinyl, and fiberglass debris. This fill material is a result of past activity at the site and is not found in the off-site areas. Ground-water monitoring has shown considerable contamination of volatile and semivolatile organic compounds and petroleum hydrocarbons. Surface water in the vicinity of the site has shown minimal contamination (E.C. Jordan Co., 1987).

Use of the Imperial Oil Co./Champion Chemical Company facility dates back to approximately 1912, when a factory that produced tomato paste and ketchup was established at the site. Near the end of World War I the facility was converted to a chemical processing plant, producing arsenic acid and calcium arsenate as well as artificial flavors and fragrances. In the late 1940s the plant was purchased by Champion Chemical Company and was used as an oil reclamation facility. Since 1968, Imperial Oil Company has leased the site from Champion Chemical Company, using the facility for blending and repackaging oils.

No process wastewater is presently generated at the facility. Site precipitation runoff is reportedly treated by oil-water separators, with the oil being collected for reuse. Waste disposal practices prior to the 1950s are generally unknown, although it has been reported that an oil-settling lagoon was once located at the rear (north corner) of the Imperial Oil Company property. At approximately the same time, large piles of oil-saturated soil or filter cake clay are alleged to have been stored at the site. Most of this material was subsequently sent off-site for disposal. The remains of one of these piles is still located along the northwest fence line at the rear of the property. This is referred to as the waste filter cake pile or waste pile. An old storage tank that contains an oily sludge of unknown origin is still present at the northern corner of the property. This is referred to as the abandoned storage tank. In addition to the plant areas, two adjacent but off-site areas north of the facility were allegedly used as dump sites for waste oil and waste sludge material.

As a result of past waste handling practices at this facility, surface and subsurface soils and ground water have been contaminated with organic chemicals, including petroleum hydrocarbons, metals, and polychlorinated biphenyls (PCBs, also known by their trade name, Aroclor, which is a mixture of PCB congeners). Potential sources of additional contamination, such as the waste pile of oil-saturated filter cake clay, still exist on the site.

Previous environmental investigations at the site included the installation of 14 groundwater monitoring wells and the excavation of six test pits. NJ DEP, with support of the U.S. EPA, is presently performing a remedial investigation and feasibility study (RI/FS) under the auspices of the Federal Superfund program to investigate and resolve the environmental contamination problems associated with the Imperial Oil site. Additional information will be available when the study is completed.

5.2 WASTE CHARACTERISTICS

Environmental media, such as surface soil, subsurface soil, and ground water, were sampled and analyzed by E.C. Jordan Company during the Phase 1 sampling as a preliminary chemical characterization of the Imperial Oil site (E.C. Jordan Co., 1987). Contaminants at the site included PCBs, metals, and organic chemicals, mainly petroleum hydrocarbons. These contaminants are thought to be primarily from previous oil recycling operations at the site, but may also be from other past activities at the facility.

The waste pile contains filter cake material contaminated with waste oil filtrate residues. The abandoned storage tank contains an oily sludge. Each is expected to contain fairly uniform contaminant levels. The two other contaminated areas of the site, Off-Site Areas One and Two, contain soil contaminated with either waste oil or oily filter cake material similar to that in the waste pile. The two off-site areas contain scattered "hot spots" of contamination that appear to decrease in intensity with depth. At several locations, these "hot spots" extend to a soil depth of 3 feet or more (PRC, 1988a). As a result of this study, Off-Site Area Two was eliminated from further study, as it was very similar in chemical composition to Off-Site Area One but less accessible.

U.S. EPA contractors visited the Imperial Oil Company/Champion Chemical Company site in May 1988 to survey and sample four possible site locations that were being considered for treatment during the Soliditech demonstration (PRC, 1988a). The samples were analyzed to determine contaminant levels at each of the locations, to evaluate their suitability for treatment. Additional sample material was also provided to allow Soliditech to test its process on actual site material and formulate appropriate treatment mixtures. In addition, the raw and treated waste from the site was provided to the analytical laboratory to determine potential analytical problems.

Additional laboratory cleanup procedures were required for all samples due to the high levels of oil and grease. Diagnosis of this problem saved considerable time when the demonstration samples were analyzed by the laboratory. (Data from the treatability study are included as Appendix C.)

The areas of the site selected for the demonstration contain various types and concentrations of chemical contamination. PCBs of concern in these areas include Aroclors 1242, 1248, and 1260. Metals found at one or more of these areas include arsenic, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, thallium, and zinc. High levels of an organic mixture identified as petroleum hydrocarbons were identified in all areas. Low levels of volatile organic chemicals (VOCs) have also been detected at the site. VOCs found in samples from these areas include benzene, ethylbenzene, tetrachloroethene, toluene, trichloroethene, 2-butanone, 2-hexanone, 4-methyl-2-pentanone, and total xylenes. Semivolatile organic chemicals (SVOCs) found in samples from these areas include bis(2-ethylhexyl)phthalate, butylbenzylphthatate, di-n-octylphthalate, 4-bromophenyl phenyl ether, and 2-methylnaphthanene (Radian, 1988 and Appendix A). This data is presented in Section 7.

5.3 DEMONSTRATION PREPARATION

Demonstration preparation included preparing a Demonstration Plan and a Health and Safety Plan, informing the public of a planned visitor's day, setting up an office trailer with electrical and phone service, mobilizing earth moving equipment, preparing decontamination zones and areas, constructing the large plywood forms to hold the treated waste, setting up sanitary facilities, making arrangements with the Imperial Oil Company to operate at their facility, and holding health and safety briefings prior to and during the demonstration. Established health and safety procedures were followed.

5.4 SAMPLING PROGRAM

The objective of the sampling program was to develop and implement sampling strategies that produce scientifically valid data useful for evaluating treated waste produced by the Soliditech process. The sampling protocols were designed to provide statistical comparisons of the physical and chemical characteristics of the contaminated wastes before and after treatment. These statistical comparisons provide the basis for determining the success of the Soliditech process at the Imperial Oil Company/Champion Chemical Company site. This section describes the waste material collection locations, sampling schedule, sample recovery procedures, and analytical procedures. Table 10 shows the numbers and sizes of samples collected for testing during the demonstration.

5.4.1 Waste Material Collection Locations

The waste material collection areas chosen for the demonstration at the Imperial Oil site are shown in Figures 2 and 3. These locations represent areas contaminated with metals, VOCs, SVOCs, petroleum hydrocarbons, and PCBs. Preliminary sampling during site screening indicated that the contaminated soil from Off-Site Area One to be used for treatment could be removed from a soil depth of up to 30 inches, compatible with the backhoe used to excavate the soil for treatment. A backhoe was also used to collect waste filter cake from the waste pile. The sludge from the abandoned storage tank was collected by hand, using a bucket. The sludge was mixed with waste pile material in the Soliditech mixer before treatment.

5.4.2 Sampling Schedule

A complex array of samples was collected to facilitate an effective evaluation of the process. Samples of contaminated waste were collected immediately prior to treatment (pretreatment samples). Process reagent and proprietary additives were used to treat a test run of

TABLE 10

SAMPLING REQUIREMENTS

PRETREATMENT WASTE SAMPLES

Parameter		Number of Samples Collected				Containers per Sample	Total Containers Used	Containers		Sampling Device	Preservation Requirements	Holding Time
		Reps (a)	Areas	Reserve (b)	Total			Type (c)	Size			
Leaching Tests	(d)	3	3	1	12	2	24	Glass	1000 mL	Scoop	Cool, 4° C	Not specified
Chemical Tests	(e)	3	3	1	12	1	12	Glass	1000 mL	Scoop	Cool, 4° C	(f)
	(g)	3	3	1	12	1	12	Glass	250 mL	Scoop	Cool, 4° C	Analyze within 14 days
Engineering/Geotechnical Tests												
	(h)	3	3	1	12	1	12	Glass	500 mL	Scoop	Seal	Not specified
	(i)	3	3	1	12	1	12	Shelby tube	3" x 12"	Shelby tube	Seal and prevent breaking	Not specified

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Notes:

(a) Reps = replicates.
(b) Reserve (contingency) samples per area.
(c) All glass jars had teflon-lined closures.
(d) Toxicity Leaching Procedure Test (TCLP), Batch Extraction Test (BET), and Extraction Procedure Toxicity Test (EP).
(e) pH, Eh, Loss on Ignition, Acid Neutralization Capacity, Oil and Grease, Metals, SVOCs, and PCBs.
(f) Analyze for pH and Eh as soon as possible.
28 days for Mercury, and Oil and Grease.
6 months for all other metals.
Extract for PCBs and SVOCs within 14 days and analyze SVOCs extract in 30 days and PCBs extract in 40 days.
Not specified for Loss on Ignition or Acid Neutralization Capacity.
(g) VOCs.
(h) Particle size and water content.
(i) Bulk density.

TABLE 10 (Continued)

SAMPLING REQUIREMENTS

REAGENT MIX SAMPLES

Parameter		Number of Samples Collected				Containers per Sample	Total Containers Used	Containers		Sampling Device	Preservation Requirements	Holding Time
		Reps (a)	Areas	Reserve (b)	Total			Type (c)	Size			
Leaching Tests	(c)	3	1	1	4	2	8	Mold	3" x 6"	Dipper	Cool, 4° C	Not specified
Chemical Tests	(d)	3	1	1	4	1	4	Mold	3" x 6"	Dipper	Cool, 4° C	(e)

Notes:

(a) Reps = replicates.
(b) Reserve (contingency) samples per area.
(c) TCLP, BET, and EP.
(d) pH, Acid Neutralization Capacity, Metals, SVOCs, and PCBs.
(e) Analyze for pH and Eh as soon as possible.
28 days for Mercury, and Oil and Grease.
6 months for all other metals.
Extract for PCBs and SVOCs within 14 days. Analyze SVOC extract in 30 days and PCB extract in 40 days.
Not specified for Loss on Ignition or Acid Neutralization Capacity.

TABLE 10 (Continued)

SAMPLING REQUIREMENTS

SOLIDIFIED WASTE SAMPLES

		Number of Samples Collected				Containers per Sample	Total Containers Used	Containers				
Parameter		Reps (a)	Areas	Reserve (b)	Total			Type (c)	Size	Sampling Device	Preservation Requirements	Holding Time
Leaching Tests	(c)	3	3	1	12	2	24	Mold	3" x 6"	Dipper	Cool, 4° C	Not specified
	(d)	3	3	2	15	1	15	Mold	2.5 cm x 4.8 cm	Dipper	Seal and prevent breaking.	Not specified
Chemical Tests	(e)	3	3	1	12	1	12	Mold	3" x 6"	Dipper	Cool, 4° C	(f)
Engineering/Geotechnical Tests												
	(g)	3	3	2	15	2	30	Mold	3" x 6"	Dipper	Seal and prevent breaking	Not specified
	(h)	3	3	2	15	2	30	Mold	4.5 cm x 7.4 cm	Dipper	Seal and prevent breaking	Not specified
	(i)	3	3	1	12	1	12	Mold	3" x 3"	Dipper	Seal and prevent breaking	Not specified

Notes:

(a) Reps = replicates.
(b) Reserve (contingency) samples per area.
(c) TCLP, BET, and EP.
(d) American Nuclear Society Test 16.1 (ANS 16.1)
(e) pH, Eh, Loss on Ignition, Acid Neutralization Capacity, Oil and Grease, Metals, VOCs, SVOCs, and PCBs.
(f) Analyze for pH and Eh as soon as possible.
Analyze for volatile organics within 14 days.
28 days for Mercury, and Oil and Grease.
6 months for all other metals.
Extract for PCBs and SVOCs within 14 days and analyze SVOCs extract in 30 days and PCBs extract in 40 days.
Not specified for Loss on Ignition or Acid Neutralization Capacity.
(g) Water content, bulk density, and unconfined compressive strength.
(h) Wet/dry weathering and freeze/thaw weathering.
(i) Permeability.

TABLE 10 (Continued)

SAMPLING REQUIREMENTS

LONG-TERM SOLIDIFIED WASTE SAMPLES

Parameter		Number of Samples Collected					Containers per Sample	Total Containers Used	Type (c)	Containers		Preservation Requirements	Holding Time
		Reps (a)	Areas (b)	Reserve (c)	Times (d)	Total				Size	Sampling Device		
Leaching Tests	(e)	3	3	1	4	48	1	48	Mold	3" x 6"	Dipper	Cool, 4° C	Not specified
	(f)	3 3	3 3	1 1	1 1	12 12	1 1	12 12	Mold Mold	3" x 18" 6" x 18"	Dipper	Seal and prevent breaking	Not specified
Special Tests	(g)	3	3	1	5	60	1	60	Mold	3" x 3"	Dipper	Seal and prevent breaking	Not specified

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Notes:

(a) Reps = replicates.
(b) Long-term monitoring was performed on the Off-Site Area One soil, the waste filter cake, and the waste filter cake/oily sludge mixture.
(c) Reserve (contingency) samples per area. Reserves will not be used for analyses unless original samples are lost or damaged.
(d) Samples for TCLP and EP leaching tests will be taken at 6 months, 12 months, 24 months, and 60 months.
(e) TCLP and EP.
(f) Waste Interface Leaching Test (WILT). Leachates will be collected from each of 18 molded core samples at two-week intervals for 30 weeks after the 28-day cure.
(g) Petrographic examination and air-void content.

clean sand (reagent mix). Samples of all test runs of treated material were collected after treatment (post-treatment samples). Sampling requirements were also established for a long-term monitoring program designed to evaluate chemical and physical stability over a period of 5 years.

The sampling and analysis contractor collected pretreatment samples of the contaminated Off-Site Area One soil, waste pile material, a mixture of storage tank sludge and waste pile material, clean sand used for the control test run, and residual material (also referred to as the predemonstration blank) remaining in the Soliditech mixer from previous work. (The residual material was cleaned out of the mixer as well as possible before the mixer was used.) They also collected treated slurry from each of the four treatment test runs.

5.4.2.1 Pretreatment Sampling Methods and Types

Pretreatment samples were required to establish the chemical and physical characteristics of the waste material and the variability associated with those characteristics. Triplicate samples of the pretreatment waste material were taken for each analytical parameter at each sampling area. An additional sample of each pretreatment material for each test was collected and held in reserve.

Undisturbed bulk density samples from Off-Site Area One and the waste pile were collected with Shelby Tubes prior to the excavation of material for the demonstration. Samples for bulk density determinations of the oily sludge and waste pile mixture were collected in glass jars after these materials were mixed in the mixing unit. Two sets of triplicate samples were collected for the physical tests -- one set for particle size and water content and the other set for bulk density.

Immediately prior to waste material collection, pretreatment samples were collected for chemical analyses, leaching tests, and chemical analyses of the leachate. The contaminated material for these samples was collected either from the excavation site (waste pile) or from the Soliditech mixer, after being thoroughly mixed but prior to adding any reagent or proprietary additives. Individual grab samples were taken from different parts of the waste pile or from the mixed raw material in the Soliditech mixer. These samples were transferred to individually labeled sample jars for shipment to the laboratory. One set of triplicate samples was collected for the TCLP, BET, and EP leaching tests. Two sets of triplicate samples were collected for chemical tests and analyzed for the following: pH, Eh, loss on ignition, acid neutralization capacity, oil and grease, metals, PCBs, VOCs, and SVOCs. The analytical procedures performed on each pretreatment waste material are shown in Table 11.

TABLE 11

STANDARD METHODS AND PROCEDURES OF SAMPLE ANALYSIS

Parameter	Sample Type	Method Number	Title	Method Type	Reference
ON-SITE TESTS					
Molds for Forming Concrete	S	ASTM C470	Molds for Forming Concrete Test Cylinders Vertically	Visual/Gravimetric	ASTM (ASTM, 1987)
Making and Curing Concrete	S	ASTM C31	Making and Curing Concrete Test Specimens in the Field	Specification	ASTM
Slump of Portland Cement Concrete	S	ASTM C143	Slump of Portland Cement Concrete	Vertical Distance	ASTM
Sampling Freshly Mixed Concrete	S	ASTM C172	Sampling of Freshly Mixed Concrete	Specification	ASTM
Homogeneity of Mixing	S	ASTM C136/C142	Sieve Analysis of Fine and Coarse Aggregates/Clay Lumps and Friable Particles in Aggregates	Sieve/Gravimetric	ASTM
ENGINEERING/GEOTECHNICAL TESTS					
Particle size	U	ASTM D-422	Particle Size Analysis	Sieve/hydrometer	ASTM
Water Content	U	Modified ASTM D-2216	Determination of Water Content of Soil, Rock, and Soil Aggregate Mixture	Gravimetric	ASTM
	T	TMSWC-4	Water Content	Gravimetric	TMSWC
Bulk Density	U	ASA-13-2	Bulk Density-Excavation Method	Gravimetric/ Volumetric	ASA (Page, 1982)
	T	TMSWC-2	Bulk Density Volumetric	Gravimetric	TMSWC

Notes:
S = Treated waste, while still a slurry.
U = Untreated waste.
RM = Reagent mix.
T = Treated waste.
LT = Treated waste, long-term monitoring.
L = Leachate.
LTL = Leachate, long-term monitoring.

TABLE 11 (Continued)

STANDARD METHODS AND PROCEDURES OF SAMPLE ANALYSIS

Parameter	Sample Type	Method Number	Title	Method Type	Reference
ENGINEERING/GEOTECHNICAL TESTS (Cont'd)					
Permeability	T	TMSWC-13	Falling-Head Permeability Test Using a Triaxial Cell	Volumetric	TMSWC
Unconfined Compressive Strength	T	ASTM D-1633	Compressive Strength of Molded Soil-Cement Cylinders	Stress Resistance	ASTM
Wet/Dry Weathering Test	T	TMSWC-12	Wet/Dry Weathering Test	Gravimetric	TMSWC
Freeze/Thaw Weathering Test	T	TMSWC-11	Freeze/Thaw Weathering Test	Gravimetric	TMSWC
SPECIAL TESTS					
Petrographic Examination	T/LT	ASTM C856	Petrographic Examination of Hardened Concrete	Visual/Optical	ASTM
Air Void Content	T	ASTM C457	Microscopic Determination of Air-Void and Parameters of the Air-Void System of Hardened Concrete	Microscopic	ASTM

Notes:

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U = Untreated waste.
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LT = Treated waste, long-term monitoring.
L = Leachate.
LTL = Leachate, long-term monitoring.

TABLE 11 (Continued)

STANDARD METHODS AND PROCEDURES OF SAMPLE ANALYSIS

Parameter	Sample Type	Method Number	Title	Method Type	Reference
LEACHING TESTS					
TCLP	U/RM/T/LT	TCLP	Toxicity Characteristic Leaching Procedure		TCLP (Federal Register, 1986)
ANS 16.1	T	ANS 16.1	American Nuclear Society 16.1 Test		American Nuclear Society (ANS, 1986)
BET	U/RM/T		Batch Extraction Test		(Côté, 1988)
EP	U/RM/T/LT	EPA Method 1310	Extraction Procedure Toxicity Test		SW 846 (U.S. EPA, 1986)
WILT	LT		Waste Interface Leaching Test		Comparison of Laboratory Batch Methods and Large Columns for Evaluating Leachate from Solid Waste, 1988. (Jackson, 1988)
CHEMICAL TESTS					
pH	U/RM/T	EPA Method 9045	Soil pH	Electrometric	SW 846
	L/LTL	EPA Method 9040	pH Electrometric Measurement	Electrometric	SW 846
Eh	U/T	Modified EPA Method 9045		Electrometric	SW 846
	L/LTL	ASTM D-1498	Standard Practice for Oxidation-Reduction Potential of Water	Electrometric	ASTM

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T = Treated waste.
LT = Treated waste, long-term monitoring.
L = Leachate.
LTL = Leachate, long-term monitoring.

TABLE 11 (Continued)

STANDARD METHODS AND PROCEDURES OF SAMPLE ANALYSIS

Parameter	Sample Type	Method Number	Title	Method Type	Reference
CHEMICAL TESTS (Cont'd)					
TDS	L	EPA Method 160.1	Residue, Filterable	Gravimetric	EPA 600 (U.S. EPA, 1979)
Acid Neutralization Capacity	U/RM/T	TMSWC-7	Acid Neutralization Capacity	Electrometric	TMSWC (Environment Canada/U.S. EPA, no date)
TOC	L	EPA Method 415.1	Organic Carbon, Total	Combustion	EPA 600
Loss on Ignition	U/T	ASTM C114	Loss on Ignition	Gravimetric	ASTM
Oil and Grease	U/T	Modified EPA Method 3550	Sonication Extraction		SW 846
	L/3550 Extract	EPA 413.2	Oil & Grease, Total Recoverable	IR	EPA 600
Metals Sb, As, Ba, Be, Cd, Cr, Cu, Ni, Ag, Tl, Zn, Ca, Al, Pb, Na	U/RM/T	EPA Method 3050	Acid Digestion of Sediments, Sludges and Soils		SW 846
Sb, Ba, Be, Cd, Cr, Cu, Ni, Ag, Zn, Ca, Al, Na	L/LTL	EPA Method 3010	Acid Digestion of Aqueous Samples and Extracts for Total Metals Analyses by FAA or ICP Spectroscopy		SW 846
Sb, Ba, Be, Cd, Cr, Cu, Ni, Ag, Zn, Pb, Na	3050/3010 Digestates	EPA Method 6010	Inductively Coupled Plasma Atomic Emission Spectroscopy	ICP Analysis	SW 846

Notes:

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LT = Treated waste, long-term monitoring.
L = Leachate.
LTL = Leachate, long-term monitoring.

TABLE 11 (Continued)

STANDARD METHODS AND PROCEDURES OF SAMPLE ANALYSIS

Parameter	Sample Type	Method Number	Title	Method Type	Reference
CHEMICAL TESTS (Cont'd)					
Pb, Tl	L/LTL	EPA Method 3020	Acid Digestion of Aqueous Samples and Extracts for Total Metal Analysis by GFAA Spectroscopy		SW 846
Pb	3020/3050 Digestates	EPA Method 7421	Lead (Atomic Absorption, Furnace Techniques)	AAS-GF	SW 846
Tl	3020/3050 Digestates	EPA Method 7841	Thallium (Atomic Absorption, Furnace Technique)	AAS-GF	SW 846
As	L/LTL/3050 Digestates	EPA Method 7060	Arsenic (Atomic Absorption, Furnace Technique)	AAS-GF	SW 846
Se	L/LTL/3050 Digestates	EPA Method 7740	Selenium (Atomic Absorption, Furnace Technique)	AAS-GF	SW 846
Hg	U/RM/T	EPA Method 7471	Mercury in Solid or Semisolid waste (Manual Cold-Vapor Technique)	AAS-CV	SW 846
Hg	L/LTL	EPA Method 7470	Mercury in Liquid Waste (Manual Cold-Vapor Technique)	AAS-CV	SW 846
Volatile Organic Analysis	U/T	EPA Method 8240	Gas Chromatography Mass Spectrometry for Volatile Organics	GS-MS	SW 846

Notes:

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T = Treated waste.
LT = Treated waste, long-term monitoring.
L = Leachate.
LTL = Leachate, long-term monitoring.

TABLE 11 (Continued)

STANDARD METHODS AND PROCEDURES OF SAMPLE ANALYSIS

Parameter	Sample Type	Method Number	Title	Method Type	Reference
CHEMICAL TESTS (Cont'd)					
Semivolatile Compounds	L/LTL	EPA Method 3520	Continuous Liquid-Liquid Extraction		SW 846
	U	EPA Method 3540/3611	Soxhlet Extraction/ Alumina Column Cleanup and Separation of Petroleum Waste		SW 846
	3540/3620 Extracts	EPA Method 8270	GC/MS for Semivolatile Organics: Capillary Column Technique	GC/MS	SW 846
Polychlorinated Biphenyls	L/LTL	EPA Method 3520	Continuous Liquid-Liquid Extraction		SW 846
	U/RM/T	EPA Methods 3540/3620/3630	Soxhlet Extraction/Florisil® Column Cleanup/ Silica Gel Cleanup		SW 846
	3520/3540 Extracts	EPA Method 8080	Organochlorine Pesticides and PCBs	GC/ECD	SW 846
	3520/3540 Extracts	EPA Method 680 (Backup)	Determination of pesticide PCBs in Water and Soil/Sediment by GC/MS	GC/MS	EPA Method 680, 1985
	RM/T	EPA Method 3550/3611	Sonication Extraction/ Alumina Column Cleanup and Separation of Petroleum Waste		SW846

Notes:

S = Treated waste, while still a slurry.
U = Untreated waste.
RM = Reagent mix.
T = Treated waste.
LT = Treated waste, long-term monitoring.
L = Leachate.
LTL = Leachate, long-term monitoring.

The clean sand used for the reagent mix/control test run and the residual material found in the Soliditech mixer upon its arrival at the site were also sampled and analyzed for chemical constituents.

5.4.2.2 Reagent Mix Sampling Methods and Types

Before treating any waste material, Soliditech performed a test run on clean sand using Urrichem and the same proprietary additives, cement, and water to be used for the waste treatment runs. Triplicate samples of this treated sand slurry were collected and used as control samples for chemical and leaching tests. A fourth sample was collected for each sample set and held in reserve. This treated reagent mix was sampled to establish which additional chemicals were added to the contaminated wastes and to establish baseline characteristics for various physical tests. Chemical tests included analyses of pH, acid neutralization capacity, metals, PCBs, and SVOCs. The leaching tests consisted of the following: TCLP, BET, and EP Toxicity test. The analytical procedures performed on the control samples are shown in Table 11.

5.4.2.3 Post-Treatment Sampling Methods and Types

Post-treatment solidified samples were required to determine the extent to which contaminated wastes had stabilized, in terms of physical, chemical, and leaching characteristics. Post-treatment samples were collected in triplicate as a slurry and allowed to cure for a minimum of 28 days prior to analyses. An additional sample from each treatment run was collected and held in reserve. Less significant changes in the chemical and physical nature of the treated material were anticipated past the 28-day curing period.

Samples to be used for the long-term evaluation of the solidified soil were scooped out of the large plywood forms, placed in labeled sample molds, and allowed to cure on-site. The size of the solidified soil sample molds depended upon the volume needed for the chemical analysis or the sample size specified for physical testing. Both immediate and long-term sample analyses were run using the solidified slurry samples collected from the cubic-yard forms and placed into smaller molds at the time of the demonstration. The treated wastes in the cubic-yard forms were allowed to cure for 28 days before they were uncrated and prepared for long-term storage. This treated waste will remain on-site for long-term monitoring. They were placed in a closely formed stack that was wrapped in 40-mil thick high-density polyethylene (HDPE) film for protection. Periodically, the solidified waste blocks will be unwrapped and examined as part of the long-term monitoring.

5.5 PHYSICAL TESTS

A number of physical tests were performed on the samples collected during the Soliditech SITE demonstration. Table 11 lists these tests, which are described in this section.

5.5.1 On-Site Tests

The treated waste slurry was subjected to the following on-site tests:

ASTM C143: Slump of Portland Cement Concrete -- A representative sample of solidified waste was placed in a dampened slump-test mold on a flat, moist, non-absorbent surface. The mold was filled in three equal-volume layers. Then, each layer was rodded with 25 strokes of the tamping rod. After the top layer was rodded, excess concrete was struck off. The mold was immediately removed from the concrete by raising it vertically. The slump was measured by determining the vertical difference between the top of the mold and the original center of the top surface of the specimen. Each test was completed in 2.5 minutes.

ASTM C136 and C142: Homogeneity of Mixing -- The homogeneity of mixing of a test run of treated waste was determined using adaptations of ASTM Methods C136 and C142. ASTM C136 is the Standard Method for Sieve Analysis of Fine and Coarse Aggregates. ASTM C142 is the Standard Test Method for Clay Lumps and Friable Particles in Aggregates. For this test a sample of the treated waste slurry was collected immediately after treatment and sieved through three different-sized sieves. The material remaining on each of the sieves was weighed. All particles that could be broken with the fingers into fines were removed by wet sieving. The three sieves were weighed again. The weight differences were due to the fractions initially retained on each sieve that passed through the sieve after breaking with the fingers. This weight of material was designated as clay lumps or friable particles. The percentage of material retained on each sieve was also calculated to determine the particle size distribution of the aggregates. The percent of clay lumps and friable particles was then calculated.

5.5.2 Laboratory Tests

The raw and treated wastes were received by the analytical laboratory and tested for a variety of physical parameters. These test methods are described below.

ASTM D422-63: Particle Size Analysis -- The particle-size distribution of the untreated waste was determined by combined sieve and hydrometer analysis. A sieve analysis was performed on that fraction of each sample larger than 0.074 mm (retained on the No. 200 sieve). The sieve analysis consisted of passing a sample through a set of sieves and weighing the portion of material retained on each sieve. Hydrometer analysis was performed on the finer fraction (less than 0.074 mm). The hydrometer analysis is based on Stoke's Law and involves preparing a dilute suspension of fine particles in water, measuring the specific gravity of the suspension at specified time intervals, and correlating settling velocity, particle diameter, and time to determine particle-size distribution.

ASTM D2216-80: Water Content, Untreated Waste -- Water content is defined as the ratio of the weight of water retained by a solid to the weight of solids, expressed as percent. ASTM Method D2216-80 was used to determine the water content of untreated waste. Moisture was determined on a dry-weight basis by measuring the mass of water removed by drying the sample to a constant mass at 110° ± 5°C.

TMSWC-4: Water Content, Treated Waste -- TMSWC-4 was used to determine the water content of treated waste. The sample was ground to pass through an ASTM No. 10 sieve. The sample mass was measured before and after it was dried in an oven maintained at 60° ± 3°C. The dry weight must be a constant weight (mass change of less than 0.03 grams in 4 hours).

ASA-13-2: Bulk Density, Untreated Waste -- The bulk density of untreated waste was determined using the ASA-13-2 Core Method (American Society of Agronomy). A cylindrical metal sampler was pressed or driven into the soil to the desired depth and removed to preserve a known volume of sample as it existed in-situ. Bulk density was calculated based on a soil sample of known volume and its mass.

TMSWC-2: Bulk Density, Treated Waste -- The bulk density of a treated waste was determined using TMSWC-2. This test was performed after the sample cured. The bulk density was determined by weighing a cylinder of the treated waste, measuring the dimensions of the cube or cylinder, and dividing the volume into the mass.

TMSWC-13: Permeability (Falling Head) -- The permeability test for solidified waste requires a sample 7.62 centimeters (3 inches) in diameter and 7.62 centimeters high. Permeability was determined using a triaxial cell and measuring changes of water volume over time under controlled temperature and pressure. A permeability coefficient was calculated based on the linear rate of flow of a fluid through a material under a hydraulic gradient of unity.

ASTM D1633: Unconfined Compressive Strength (UCS) -- The UCS characteristics of molded treated waste cylinders were determined using strain-controlled application of an axial load. UCS is defined as the load per unit area, expressed as pounds per square inch (psi), at which an unconfined cylindrical sample of solids will fail a compression test.

TMSWC-12: Wet/Dry Weathering Test -- This test was performed using two 4.5 centimeter (cm) diameter x 7.4 cm high specimens of solidified waste. One of the specimens was the test sample, the other was the control. Each sample was removed from its mold, placed in a tared beaker, and weighed. The control was placed in a humidity chamber maintained at 22 ± 3°C, while the test specimen was dried in a vacuum oven at 60 ± 3°C for 24 hours. The dried specimen was cooled to room temperature in a desiccator. Then 230 mL of water was added to each of the sample beakers. Both samples are placed in the humidity chamber for 24 hours. The samples were then sprayed with distilled water to remove loosely attached particles from the specimens. The specimens were transferred to two, new tared beakers. The original beakers were placed in the oven to evaporate the water and dry them to a constant weight. This was repeated 11 times, or until the specimen lost its physical integrity, with the weight loss being recorded each time. The corrected relative weight loss of the test specimen was obtained by subtracting the relative weight loss of the control from the relative weight loss of the sample.

TMSWC-11: Freeze/Thaw Weathering Test -- This test was performed on two 4.5 cm diameter x 7.4 cm high specimens of solidified waste. One of these specimens was used as a control. Each sample was removed from its mold, placed in a tared beaker, and weighed. The control was placed in a humidity chamber maintained at 22 ± 3°C, while the test specimen was placed in a freezer at -20 ± 3°C for 24 hours. The specimens were removed from the freezer and moisture chamber and 230 mL of distilled water was added to each beaker. Both beakers were placed in the moisture chamber for 24 hours. The samples were then sprayed with distilled water to remove loosely attached particles from the specimens. The specimens were transferred to two, new tared beakers. The original beakers were placed in an oven to evaporate the water and dry them to a constant weight. This procedure was repeated 11 times, or until the sample lost its physical integrity, with the weight loss being recorded each time. The corrected relative weight loss of the test specimen was obtained by subtracting the relative weight loss of the control from the relative weight loss of the sample.

5.6 CHEMICAL TESTS

Chemical tests, including actual chemical analyses, and tests for chemical properties were performed on the untreated and treated waste samples collected during the Soliditech demonstration. The untreated wastes, reagent mix/control, treated wastes, and resultant leachates were analyzed for organic and inorganic constituents. The sample preparation and analytical procedures are summarized below.

SW-846 Method 9045: pH-Soil/Solid -- Equal weights of the solid and laboratory pure water were mixed to form a slurry and allowed to settle for 1 hour. The pH of the supernatant was measured el[illegible]ometrically using a combination pH electrode. For calcareous waste, the laboratory pure water was replaced by a calcium chloride solution (0.01 M).

SW-846 Method 9040: pH-Aqueous -- The pH of aqueous samples or leachates was measured electrometrically using a combination pH electrode.

SW-846 Modified Method 9045: Oxidation-Reduction Potential (Eh), Soil/Solid -- A solid/water slurry was prepared as in Method 9045. The redox potential for the supernatant was measured electrometrically using a combination oxidation-reduction electrode.

ASTM D1498-76 Oxidation-Reduction Potential (Eh), Aqueous -- The oxidation-reduction potential of an aqueous solution was measured electrometrically using a combination oxidation-reduction electrode.

U.S. EPA 160.1: Residue, Filterable (Total Dissolved Solids, TDS) -- A well-mixed leachate aliquot was filtered through a standard glass fiber filter. The filtrate was collected in a tared beaker, evaporated, and dried to a constant weight at 180°C.

TMSWC-7: Acid Neutralization Capacity -- A 150-gram sample of the untreated waste, reagent mix/control, or cured solidified waste was dried to a constant weight at 60 ± 3°C. The sample was then ground to pass an ASTM No. 100 sieve. Ten-gram aliquots of the sample were added to a series of 10 centrifuge tubes. Distilled water and 2 normal nitric acid were added to each tube in volumes that resulted in 10 different extraction fluids. The samples were shaken and then placed on a rotary extractor for 48 hours. The samples were then centrifuged. The pH of each supernate was measured and recorded. This method did not allow a complete titration of the post-treatment samples to acidic pH. The neutralization potential (expressed as percent $CaCO_3$) was determined by titrating a 2.0 gram aliquot of each post-treatment sample with 0.5 N HCl (U.S. EPA, 1978).

U.S. EPA 600 Method 415.1: Total Organic Carbon (TOC) -- An aliquot of the leachate was acidified with sulfuric acid to a pH of less than 2. An inert gas was bubbled through the sample to drive off the carbonates. Organic carbon in the sample was then converted to carbon dioxide by catalytic combustion. The carbon dioxide was measured directly by an infrared detector. TCLP and EP leachate were not analyzed for TOC. These leachates contain high acetate concentration that would adversely affect TOC data interpretation.

ASTM C114: Loss on Ignition -- The loss on ignition method is a technique for determining the total moisture and carbon content of a cementitious solid. A sample was ignited at a temperature of 950°C for 15 minutes. The percent of weight lost was calculated based on the weight of the original sample. A correction factor was applied when the sample contained a substantial quantity of sulfide.

U.S. EPA 600 Method 413.2: Oil and Grease -- The oil and grease in the untreated and solidified wastes were extracted by sonication using a modified SW-846 Method 3550. Five grams of the solid was added to 25 milliliters (mL) of freon and sonicated. The leachates were acidified with hydrochloric acid to a pH of less than 2 and extracted with multiple aliquots of freon in a separatory funnel. The extract was then taken up to 100 mL with freon. The oil and grease in the extracts were then determined by infrared spectroscopy.

SW-846 Method 8240: Volatile Organic Compounds (VOCs) -- Method 8240 in SW-846 is a gas chromatography-mass spectrometry (GC/MS) procedure used to determine the concentration of VOCs, having a boiling point below 200°C, in solid and liquid samples. Solid samples were prepared by placing 5 grams of the solid in 10 milliliters of methanol. An inert gas was bubbled through a solution containing an aliquot of the sample, at ambient temperatures. The VOCs were transferred from the aqueous phase to the vapor phase. The vapor was swept through a sorbent column, and the VOCs were absorbed. After purging was completed, the sorbent column was heated and backflushed with inert gas to desorb the components onto a gas chromatography column. The VOCs were separated by GC and detected by MS.

SW-846: Semivolatile Organic Compounds (SVOCs) -- Several methods were used for SVOC analyses, based on the form of the waste - soil, sludge, or leachate. The following describes standard U.S. EPA methods, from SW-846, for extraction and analysis of SVOCs.

1. Method 3520 is a procedure for isolating organic compounds from aqueous samples. A measured volume of sample was extracted with methylene chloride using a continuous liquid-liquid extractor. The extract was dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step to be used.
2. Method 3540 is a procedure for extracting SVOCs from solids such as soils, sludges, and wastes. The solid sample was mixed with anhydrous sodium sulfate, placed in an extraction thimble, and extracted using an appropriate solvent in a Soxhlet extractor. The extract was then dried, concentrated, and, as necessary, exchanged with a solvent compatible with the cleanup or determinative step being employed.
3. Method 3611 is a procedure for the cleanup of petroleum wastes from sample extracts. The cleanup column is packed with alumina, topped with a water

absorbent, and then loaded with the sample. A suitable solvent was used to elute the analytes while leaving the interfering compounds on the column. The eluent was then concentrated and submitted for analyses.

4. **Method 8270 is a capillary column procedure used to determine the concentration of SVOCs in sample extracts. Method 8270 was used to quantify most SVOCs that were soluble in methylene chloride, including polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamine, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols.**

SW-846: Polychlorinated Biphenyls (PCBs) -- Several methods were used for PCB analyses, based on the form of the waste -- soil, sludge, or leachate. The following describes standard U.S. EPA methods, from SW-846, for PCB extraction, screening, and analysis.

1. **Method 3520 is a procedure for isolating organic compounds from aqueous samples. A measured volume of sample was extracted with methylene chloride using a continuous liquid-liquid extractor. The extract was dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step to be used.**

2. **Method 3540 is a procedure for extracting SVOCs from solids such as soils, sludges, and wastes. The solid sample was mixed with anhydrous sodium sulfate, placed in an extraction thimble, and extracted using an appropriate solvent in a Soxhlet extractor. The extract was then dried, concentrated, and, as necessary, exchanged with a solvent compatible with the cleanup or determinative step being employed.**

3. **Method 3550 was used to extract SVOCs from the post-treatment waste to allow easier access to the samples when pH adjusting to retrieve the acid compounds. The samples were strongly alkaline and required close observation to maintain the pH required. A 30-gram sample was mixed with anhydrous sodium sulfate. This mixture was extracted three times using sonication. The extract was separated from the sample by centrifugation, and then submitted for cleanup. This method could not be performed on the reagent mix samples because of the excessive alkalinity of these samples.**

4. **Method 3620 is a procedure for the cleanup of sample extracts. The cleanup column was packed with Florisil®, topped with a water absorbent, and then loaded with the sample. A suitable solvent was used to elute the analytes of interest while leaving the interfering compounds on the column. The eluent was then concentrated and submitted for analyses or further cleanup.**

5. **Method 3630 is another procedure for the cleanup of sample extracts. This cleanup procedure was applied to the solid sample extracts after they underwent Florisil® cleanup. This procedure was used to remove pentachlorophenol from the extracts to be analyzed for PCBs. Pentachlorophenol was found to be present and to cause interferences in the analysis of PCBs in the preliminary samples. The cleanup column was packed with silica gel adsorbent, topped with water absorbent, then loaded with the sample extract. A suitable solvent was used to elute the analytes of interest while leaving the interfering compounds on the column. The eluent was then concentrated and submitted for analyses.**

6. Method 8080 specifies gas chromatographic conditions for detecting PCBs. Samples were injected into the GC column. Compounds in the GC column effluent were detected by an ECD.

7. Method 680 covers pesticides and PCBs in waters, soils, and sediments by GC/MS. It is applicable to samples containing single PCB congeners or to samples containing complex mixtures, such as Aroclors. PCBs were identified and measured as isomer groups by levels of chlorination.

   Method 680 is designated as a backup for Method 8080 to quantify PCB congeners in aqueous extracts. Unfortunately, there is no clear protocol for determining when Method 680 can effectively be used as a backup to Method 8080. The steps to be followed consist of analyzing an aqueous extract by Method 8080. If no Aroclor patterns are identified, the chromatogram is visually inspected to determine the presence of peaks that are suspected of being PCB congeners. Because the response factor cannot be easily established for peaks by GC analysis, the best judgment of the analyst is used to determine whether Method 680 should be employed. It was not necessary to use this method for the Soliditech demonstration.

SW-846: Metals -- Several methods were used for metals analyses, based on the type of metal and the form of waste -- solid, sludge, or leachate. The following describes standard U.S. EPA methods from SW 846 for digestion and analysis of metals. The appropriate digestion and analytical procedures for metals are presented in Table 12.

1. Method 3050 is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (AAS) or inductively coupled plasma emission spectroscopy (ICP). A representative sample was mixed with nitric acid (HNO_3) and refluxed. Hydrogen peroxide (H_2O_2) was then added to the digestate with gentle heating. If the sample was being prepared for graphite furnace AAS analyses, then the digestate was filtered and brought to volume with deionized water. If the sample was being prepared for ICP analysis, then hydrochloric acid (HCl) was added to the digestate. After gentle heating, the digestate was filtered and brought up to volume with deionized water.

2. Method 3010 is a digestion procedure used to prepare aqueous samples for analysis by flame AAS and ICP. The sample was mixed with HNO_3 and allowed to reflux in a covered Griffin beaker, followed by hydrochloric acid to dissolve any precipitate or residue resulting from evaporation. The sample was then filtered and brought up to volume using deionized water.

3. Method 3020 is a digestion procedure used to prepare aqueous samples for lead and thallium analysis by graphite furnace AAS. The sample was mixed with HNO_3 and allowed to reflux in a covered Griffin beaker. A small volume of deionized water was added with continued warming to dissolve any residue resulting from evaporation. The digestate was then reconstituted with deionized water.

TABLE 12

DIGESTION AND ANALYTICAL TECHNIQUES FOR ELEMENTAL ANALYSIS

Analyte	Technique	Matrix Type	Method Number
Sb, As, Be, Cd, Cr, Cu, Pb, Ni, Se, Ag, Tl, Zn, Na	D	Solid	3050
Sb, Be, Cd, Cr, Cu, Ni, Ag, Zn, Na	D	Leachate	3010
Pb, Tl	D	Leachate	3020
As, Se	D	Leachate	7060/7740
Sb, Be, Cr, Cu, Ni, Ag, Zn, Pb, Na	A	Solid/Leachate	6010
As	A	Solid/Leachate	7060
Pb	A	Leachate	7421
Se	A	Solid/Leachate	7740
Tl	A	Solid/Leachate	7841
Hg	D & A	Leachate	7470
Hg	D & A	Solid	7471

Notes:

D = Digestion
A = Analytical

4. Methods 7060 and 7740 describe the digestion procedure used to prepare aqueous samples for arsenic and selenium analysis by graphite furnace AAS. The sample was mixed with hydrogen peroxide and nitric acid and allowed to reflux in a covered Griffin beaker for one hour. The sample was then brought back to volume using deionized water.

5. Method 6010 describes the simultaneous, or sequential, determination of elements using ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element specific atomic-line emission spectra are produced, dispersed by a grating spectrometer and monitored for intensity by photomultiplier tubes.

6. Methods 7060, 7421, 7740, and 7841 are graphite furnace AAS methods for determining arsenic, lead, selenium, and thallium, respectively. Following sample digestion, an aliquot of sample was placed in a graphite tube in the furnace, evaporated to dryness, charred, and atomized. The materials analyzed were then placed in the light path of an atomic absorption spectrophotometer.

7. Method 7470 is a cold-vapor atomic absorption (CVAAS) procedure for determining the concentration of mercury in mobility-procedure extracts. Method 7471 is prescribed for solid and sludge-type wastes. A sample aliquot was acidified. Potassium permanganate was added to maintain oxidizing conditions. Potassium persulfate was added, and the sample was heated in a water bath. After cooling, mercury in the sample was reduced to the elemental state (with the addition of hydroxylamine hydrochloride and stannous sulfate) and aerated from solution in a closed system. The mercury vapor passed through a cell positioned in the light path of an atomic absorption spectrophotometer.

Please note that digestion Methods 3010 and 3050 do not contain antimony (Sb) or silver (Ag) in their target analyte list. The sample matrices to be analyzed are leachate, soils, and solids and are not covered by the methods listed for these elements (SW-846 Methods 3005 and 7040). Radian maintained laboratory control charts that show that the average blank spike recoveries for Ag and Sb using Methods 3010 and 3050 in their laboratories were within recovery limits. The median recoveries are 90 percent for both Ag and Sb. Methods 3010 and 3050 were acceptable and were used to digest leachate, soil and solid samples for Ag and Sb.

5.7 LEACHING TESTS

Leaching/extraction were performed on untreated and treated waste samples collected during the Soliditech demonstration. These tests were performed using both destructive methods (TCLP, EP Toxicity, and BET) and non-destructive methods (ANS 16.1 and WILT). Destructive methods crush or grind the samples prior to testing destroying the physical integrity of the solidified waste samples. Non-destructive methods were applied to intact cast cylinders. The procedures are briefly summarized below.

Federal Register, 1986: Toxicity Characteristics Leaching Procedure (TCLP) Test -- The TCLP test was designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic wastes. The sample was prepared by crushing the waste. The leaching procedure involved a solid-to-liquid ratio of 1:20, maintained at a specified pH that was agitated in a rotary extractor for 18 ± 2 hours, and filtered through a 0.6-mm to 0.8-mm glass fiber filter. The pH and type of leaching medium used depend on the alkalinity of the solid phase of the waste. Preparing treated waste samples for the TCLP extraction of VOCs was a critical concern. A validated laboratory procedure for preparing these samples was not available. Special care was taken to minimize the chance for VOC releases into the atmosphere. The sample was placed in the zero headspace extractor (ZHE) immediately after pulverization.

U.S. EPA Method 1310: EP Toxicity Test -- The extraction procedure (EP) toxicity test is used to determine whether a waste exhibits the RCRA characteristic of toxicity. This test may also be used to simulate the leaching that a waste would undergo in a sanitary landfill. The sample was prepared by crushing it to pass through a 9.5 mm sieve. A single batch of material was extracted at a solid-to-liquid ratio of 1:20. The extract was maintained at pH 5.9 ± 0.2, using 0.5 normal acetic acid, and stirred or tumbled for 24 hours. The extract was filtered through a 0.45 μm membrane filter. The leachate was analyzed for the standard list of EP parameters using appropriate analytical methods.

Côté, 1988: Batch Extraction Test (BET) -- The BET is a modification of TMSWC-6, developed by Dr. P. Côté of the Wastewater Technology Center and Alberta Environmental Center (Côté, 1988). After the 28-day curing period, the treated waste was crushed to pass an ASTM No. 100 sieve and the water content was determined. Samples of three solid-to-liquid ratios (1:4, 1:20, and 1:100) were prepared for each treated waste. The samples were extracted on a rotary extractor for 7 days. The total dissolved solids and the soluble fraction of waste were determined on the 4:1 liquid-to-solid extract. The pH of each extract was measured, the sample filtered, and the resulting leachate submitted for chemical analyses.

American Nuclear Society: 16.1 Test (ANS 16.1) -- The 28-day modification of ANS 16.1 for solidified wastes was used to approximate leaching from intact (not crushed) solidified waste by rapidly flowing ground water. Samples were leached without agitation using demineralized water. The demineralized water had an electrical conductivity less than 5 μmho/cm at 25°C, and a total organic carbon concentration of less than 3 milligrams per liter. The samples were totally submerged in the water for a specified test period, then the leachate was collected and set aside for analyses. The sample was then re-extracted in another aliquot of demineralized water. This cycle was repeated until five leachates were obtained for each sample.

Jackson, 1988: Waste Interface Leaching Test (WILT) -- The WILT was designed to evaluate the long-term leaching behavior of intact (not crushed) solidified waste. Two sizes of cores (3-

inch diameter by 18-inch length, and 6-inch diameter by 18-inch length) of the treated wastes were prepared in triplicate. Each of these cores was placed into individual leaching columns, and the annular space between the core and container was filled with acid-washed sand. A 1-inch layer of acid-washed sand was added to the top of the columns to disperse the leaching fluid uniformly over the cross-sectional area of the treated waste.

A 0.25-inch diameter tygon tube was connected to the hose-bib at the base of each column with the opposite end fitted to a three-way stopcock. A second tygon tube was connected to the stopcock and a fitting on sealed Tedlar® bags. This arrangement allows leachate from the columns to flow into the Tedlar® bags and facilitates sampling of leachate in the bags without exposing it to the atmosphere.

Leaching fluid (distilled water) was supplied through the bottom of each column through tubing connected to a water reservoir. Deionized water was added to fully saturate each column to the surface level of the sand packing. The columns remained saturated until leachate was collected at predetermined biweekly or monthly intervals. Biweekly leachate collection and analysis was performed for a period of 2 months, followed by biweekly leachate collection with monthly leachate analysis for 4 additional months. After six months, leachates will continue to be collected biweekly but will be analyzed every other month.

5.8 QUALITY ASSURANCE AND QUALITY CONTROL SUMMARY

The Soliditech Demonstration Plan included a Quality Assurance Project Plan (QAPP) that detailed quality assurance and quality control (QA/QC) procedures for the demonstration sampling and analysis activities. These QA/QC procedures included the following:

- QC Check Samples -- Standard samples of known analyte concentrations.
- Laboratory Blank Samples -- The laboratory analyzed calibration or reagent blanks at the beginning of each analytical run and every 10 samples thereafter. Calibration blanks consisted of deionized water and were not taken through any sample preparation steps. Reagent blanks consisted of deionized (or organic-free) water taken through all sample preparation steps, including adding reagent and digestion/extraction procedures.
- Calibration Check Compounds -- Standards used for ongoing calibration verification.
- Spiked Samples -- A small subset of the samples were spiked with known concentrations of either reference materials or surrogate standards and taken through the sample preparation process. Spiked samples allowed the laboratory to assess the efficiency of extraction processes, the accuracy of the analyses, and possible matrix effects.

- Duplicate samples -- These samples were analyzed at a 10 percent frequency. Duplicate sample analyses provided a measure of sample variability.
- Replicate samples -- Sample extracts, digestates, or leachates were analyzed in replicate at a 10 percent frequency. Replicate sample analyses provided a measure of analytical variability.

U.S. EPA performed both a field audit during the demonstration and a laboratory audit to ensure that all QA/QC procedures were being followed. Both audits found the sampling and analysis activities satisfactory.

Overall, the quality control results for the Soliditech program were excellent. A few problems were noted and are listed below:

- Recoveries of matrix spike were outside of the acceptance criteria for selenium and thallium for both pre- and post-treatment matrices.
- Holding times were exceeded for the extraction of pre-treatment TCLP leachates analyzed for VOCs by Method 8240.
- Poor recoveries were recorded for Method 8270 acid fraction matrix spike and surrogate compounds for post-treatment samples.

These problems are discussed in detail in Appendix B.

6.0 FIELD ACTIVITIES

The Soliditech demonstration field activities consisted of the collection of waste materials, the treatment of the waste materials, and the discharge and sampling of the treated waste. Section 6.1 discusses collection, Section 6.2 discusses treatment, and Section 6.3 discusses sampling. The chronology of the major demonstration events is shown in Table 13.

6.1 WASTE MATERIAL COLLECTION

Waste material was collected from three areas of the Imperial Oil Company/Champion Chemical Company Superfund site. These areas were identified as the abandoned storage tank, the waste pile, and Off-Site Area One. The locations of these areas are shown in Figures 2 and 3.

6.1.1 Summary of Operations

This section details the waste material collection from each of the three areas previously chosen for treatment.

Abandoned storage tank. A catch basin was excavated next to the tank manhole and lined with plastic sheeting to contain any releases of waste. The manhole located in the side of the tank was then opened, and the oily sludge in the tank was removed by bucket from the tank and placed in 55-gallon drums. Three full drums of this material were collected, sealed, weighed, and staged for later treatment. Due to the liquid nature of this waste material, Soliditech blended the oily waste material from this tank with solid filter cake material from the waste pile. This blending was performed in the mixer immediately prior to treatment.

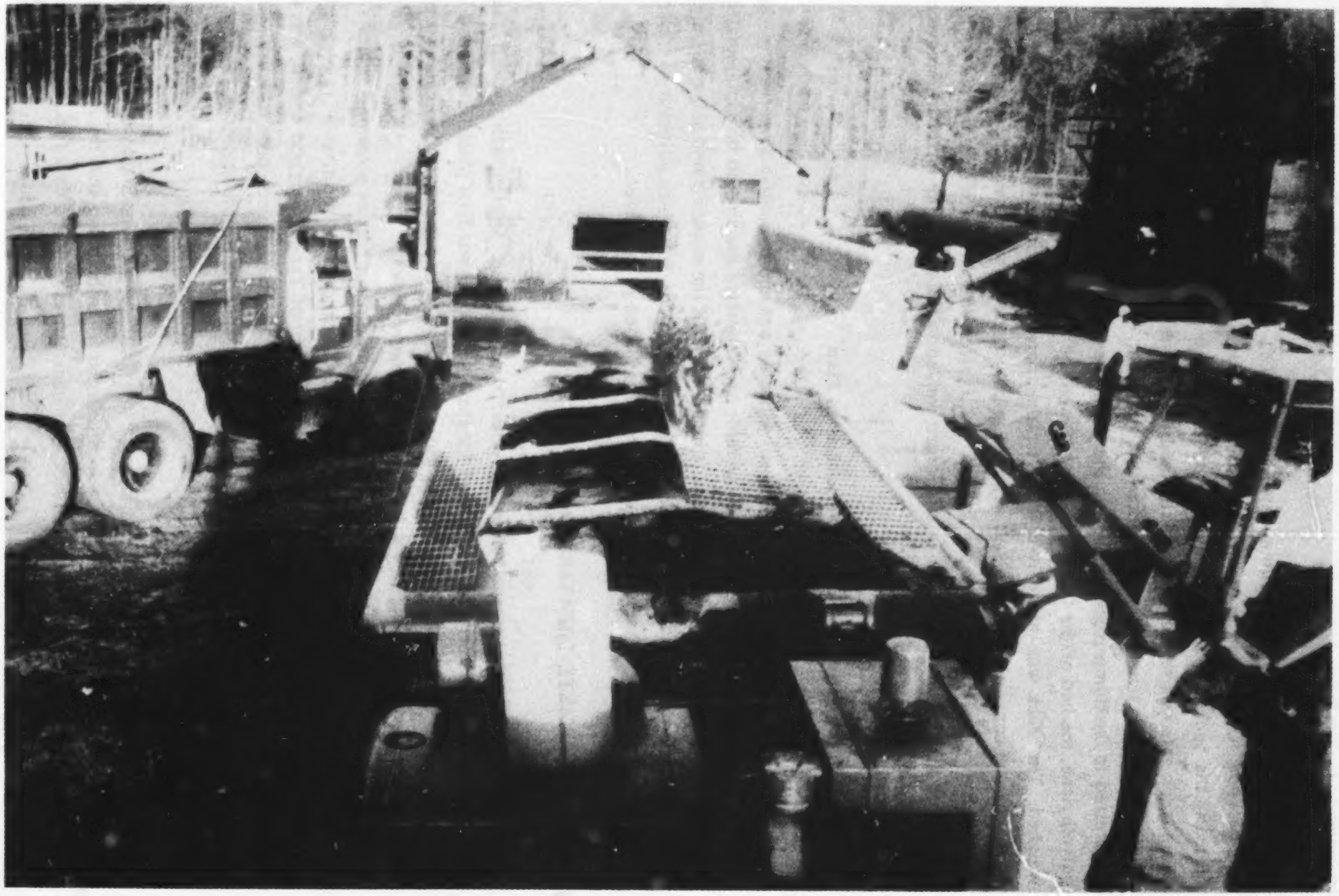
Waste Pile. Three 55-gallon drums were filled with filter cake material from the waste pile, using a backhoe. These drums were weighed to obtain the density of this waste and the results used to calibrate the bucket of the front-end loader for subsequent transfer of this waste to the mixer. Once the front-end loader was calibrated, the filter cake material was collected from the exposed face of the waste pile and directly transferred by the front-end loader into the Soliditech mixer, through a 4-inch by 4-inch screen welded to the top of the large mixer. Figure 5 shows the 4-inch by 4-inch mesh screen on top of the mixer that was used to remove large debris from the waste material prior to treatment. No attempts were made to collect this material from the most contaminated areas of the waste pile.

TABLE 13

SOLIDITECH DEMONSTRATION CHRONOLOGY

Site Preparation	
Obtain sampling and analysis equipment	**December 1 and 2, 1988**
Establish health and safety zones	**December 2, 1988**
Office trailer set-up	**December 2 and 5, 1988**
Setup decontamination areas	**December 5 and 6, 1988**
Accommodation of Soliditech equipment	**December 5 and 6, 1988**
Preparation for visitor's day	**December 6, 1988**
Waste collection	**December 6 and 7, 1988**
Visitor's day	**December 7, 1988**
Operations	
Arrival of Soliditech equipment	December 3, 1988
Set-up of the Soliditech equipment	December 5 and 6, 1988
Waste material collection	
Off-Site Area One	December 6, 1988
Filter cake/oily sludge	December 6 and 7, 1988
Filter cake	December 7, 1988
Pretreatment sample collection	
Off-Site Area One	December 5, 1988
Filter cake/oily sludge	December 7, 1988
Filter cake	December 7, 1988
Waste treatment and post-treatment sampling	
Reagent mix/control run	December 6, 1988
Filter cake/oily sludge	December 7, 1988
Filter cake	December 7, 1988
Off-Site Area One	December 8, 1988
Clean-up activities	December 8 through 14, 1988
Sample Collection and Preparation of Long-Term Monitoring Area	
Collection of treated waste samples	January 9, 1989
Removal of forms from treated waste monoliths	January 9, 1989
Placement of monoliths in long-term monitoring location	January 10, 1989

FIGURE 5
WASTE CONTAINED IN 10-CUBIC-YARD SOLIDITECH MIXER



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Off-Site Area One. Contaminated soil from Off-Site Area One was collected by means of a backhoe. During collection large pieces of earth and roots were removed. The soil was placed in the bucket of a tracked front-end loader and transported to a nearby dump truck. The dump truck gate was sealed with caulking and its bed was lined with two layers of 8-mil polyethylene. The truck was equipped with a bed cover. When the contaminated soil was transferred to the dump truck, the soil in the truck bed was covered with the two sheets of polyethylene, the bed of the truck was covered, and the truck was driven to a scale where it was weighed. The truck was then driven to the treatment area where it remained overnight. Prior to treatment, the contaminated soil, still wrapped in polyethylene, was deposited next to the Soliditech mixer. Prior to treatment this soil was transferred to the mixer via a front-end loader, and passed through the 4-inch by 4-inch screen welded to the top of the mixer to remove any large pieces of debris.

6.1.2 Deviations from the Demonstration Plan

The amount of Off-Site Area One soil collected was approximately 57 percent of the amount planned, because the equipment operator overestimated the amount of contaminated soil that he collected. The discrepancy was not discovered until the contaminated soil was weighed and found to be three- to four-cubic-yards less than planned. Soliditech adjusted its treatment formulation based upon the weight of waste that was loaded in the mixer.

6.2 WASTE TREATMENT

Both mixers were scraped and steam-cleaned prior to use. During each test run, the contaminated waste material (or clean sand for the control run) was first placed in the Soliditech mixer. As mentioned in Section 6.1.1, the waste pile material and Off-Site Area One soil were first screened through a 4-inch by 4-inch screen to remove any rocks, roots, or other large debris. The oily sludge and the sand for the control run did not require screening. Next, predetermined amounts of Urrichem reagent, water, and proprietary additives were blended with the contaminated waste. Finally, a predetermined amount of Type II portland cement was added, using the front-end loader with a calibrated bucket, and treatment commenced. Figure 5 shows the addition of cement to the large Soliditech mixer. During the treatment process, mixing was continually evaluated by Soliditech personnel.

After approximately 40 to 60 minutes, when Soliditech personnel determined that each batch was adequately mixed, the slurry was discharged from the mixer into the reinforced one-cubic yard plywood forms. When one form was filled, it was removed by a forklift and another empty form moved into place and filled. This process continued until the mixer was emptied. Figure 6 shows the demonstration in progress, with the large Soliditech mixer discharging into a plywood form. (The small Soliditech mixer can be seen on to the right of the photograph on the truck bed, the Soliditech cement storage hopper can be seen to the left of the photograph, and the waste pile can be seen in the center of the photograph behind the large Soliditech mixer.)

The treated waste in one form from each of the four test runs was allowed to cure indoors at a temperature of approximately 50 to 70°F for 24 hours (see Section 6.3.2). All other treated waste cured outdoors at ambient temperatures (25 to 40°F). All treated waste in the plywood forms completed the prescribed 28-day cure in an unheated warehouse. Although 11 of the blocks of treated waste were allowed to cure outdoors, at times when temperatures were below freezing, the treated waste had already hardened prior to being subjected to sub-freezing temperatures.

After completion of the curing time, the treated waste was removed from the warehouse, the plywood sides of the forms were removed, the treated waste was organized into a stack and the stack was wrapped in 40 mil high-density polyethylene (HDPE) film. The stack will remain on site for long-term study and observation.

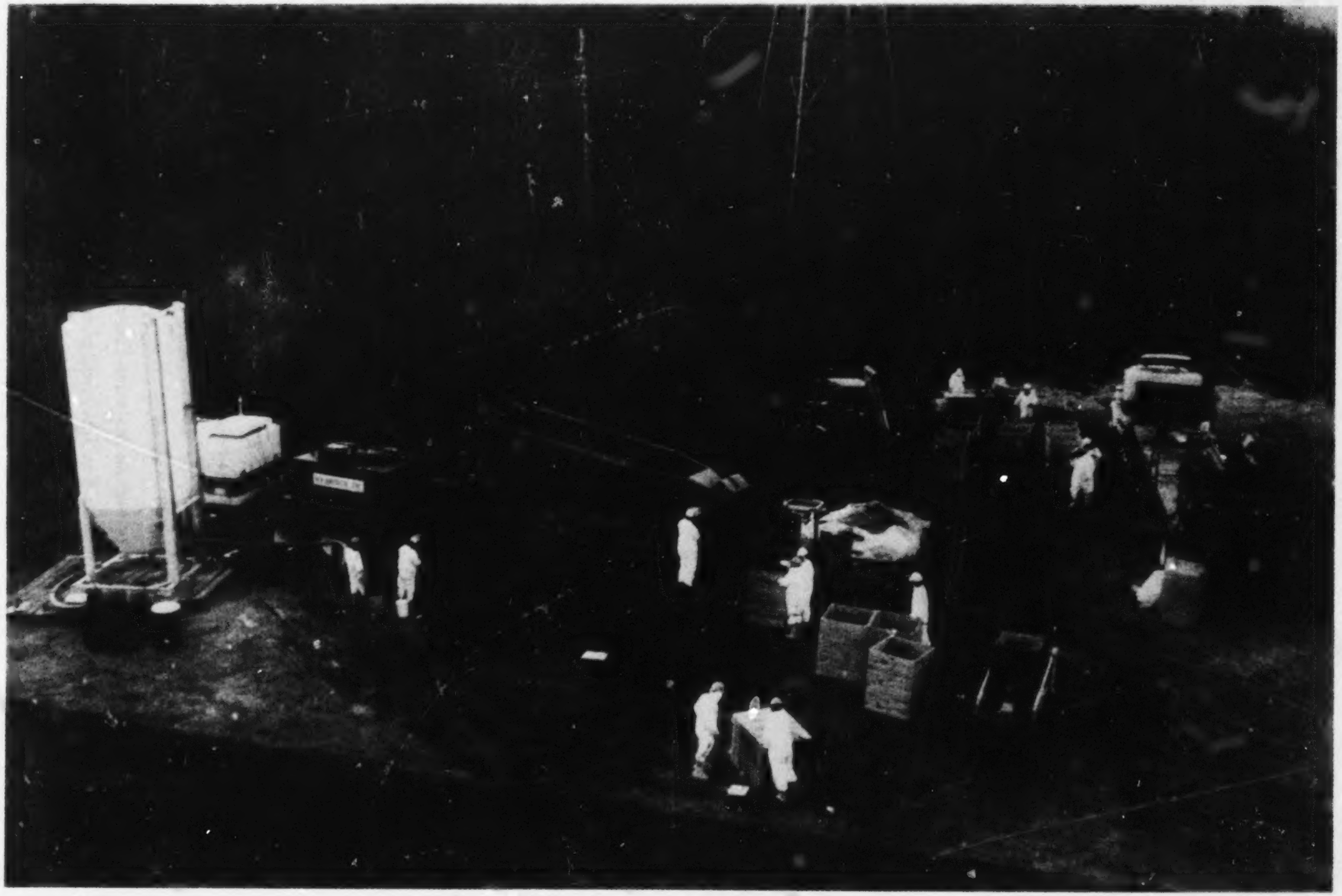
6.2.1 Summary of Test Runs

The first test run was performed on a batch of clean sand rather than contaminated waste material. This test run was used as a control run and is referred to as the reagent mix or control test run. Due to the small amount (800 pounds) of sand being solidified during this run, the smaller of the two Soliditech mixers was used. The sand was dumped directly into the Soliditech mixer from the sand bags. As detailed above, water, reagent, proprietary additives, and cement were added and thoroughly mixed with the sand. The mixture was blended for approximately 40 minutes.

The second test run was performed on the mixture of oily sludge and filter cake material, blended at one part oily sludge to two parts waste filter cake material. Approximately 2 cubic-yards of this waste were treated. Once the two wastes were thoroughly mixed in the large Soliditech mixer, water, reagent, proprietary additives, and cement were added and thoroughly mixed with the waste. This mixture was blended for approximately 45 minutes.

The third test run was performed on the same filter cake material but without the addition of oily sludge. Approximately 5 cubic-yards of this waste were treated. Once the waste

FIGURE 6
DISCHARGING OF TREATED WASTE INTO PLYWOOD FORMS



was added to the large Soliditech mixer, water, reagent, proprietary additives, and cement were added and thoroughly mixed with the waste. This mixture was blended for approximately 45 minutes.

The fourth test run was performed on the Off-Site Area One soil. Approximately 4 cubic-yards of this waste were treated. Once the waste was added to the large Soliditech mixer, water, reagent, proprietary additives, and cement were added and thoroughly mixed with the waste. This mixture was blended for approximately one hour.

6.2.2 Deviations from the Demonstration Plan

Overall, the waste treatment phase of the demonstration was considered to be a success. There were several deviations from the Demonstration Plan. These deviations are discussed below.

In the first test run of waste material (filter cake/oily sludge mixture), material was trapped at one end of the mixer and not completely blended. This was discovered as the treated waste was discharged from the mixer. This was probably due to lack of operator experience with the mixer and with this waste type. It could have been remedied by more vigorous end-to-end agitation of the mixer. Since the trapped material was present in the solidified matrix, it will be evaluated with the samples from this run.

The waste treatment test runs were performed in a different order than originally planned. The reagent mix (clean sand and all Soliditech's reagent and proprietary additives) was treated first, as planned. Due to the late arrival of the front-end loader required to collect the Off-Site Area One soil, the mixture of oily sludge and filter cake material was treated second, rather than last. The filter cake material was treated third, as planned, and the Off-Site Area One soil was treated last. This change was inconsequential and did not affect other operating plans.

The large Soliditech mixer, although cleaned prior to the SITE demonstration, contained some residual material. Soliditech personnel scraped as much of this material out of the mixer as possible and then steam cleaned the mixer prior to its use for the demonstration. A sample of the material scraped from the mixer was collected and chemically analyzed to determine whether it could contaminate the demonstration samples. From these results it was determined that any residual material could not have contributed to the contamination found in the treated waste. These analytical results are presented in Appendix A.

After treatment of the filter cake/oily sludge mixture, the large Soliditech mixer was scraped and steam-cleaned, as specified in the Demonstration Plan. After treatment of the filter cake material alone, however, it was determined that it was not necessary to thoroughly clean the mixer, since the contaminated Off-Site Area One soil that was to be treated next would be sampled directly from the mixer. Any residual material from the previous batch would be thus mixed with the soil, sampled, and treated as part of the contaminated soil. This procedure was more representative of actual conditions, when, to save operating time, the mixer would not be cleaned between each batch.

6.3 SAMPLING FOR PROCESS EVALUATION

To evaluate the Soliditech process, samples of waste material were collected before and after treatment. These samples were analyzed by chemical and physical tests and were exposed to various extraction liquids in five separate leaching tests. The liquid extracts from these leaching tests were chemically analyzed. A reagent mix or control test run was also performed, using clean sand in place of the waste material.

6.3.1 Pretreatment Sampling Procedures

Prior to each test run, samples of each waste material were collected, in triplicate, in glass jars or Shelby tubes. Shelby tube samples for bulk density determinations on the solid waste materials were collected from the actual location of the waste material prior to waste collection. All pretreatment samples from filter cake/oily sludge mixture, including the bulk density samples, were collected in glass jars directly from the mixer after blending but prior to treatment. All pretreatment samples from the waste pile were collected from several locations in the waste pile, which was considered to be uniform in composition. With the exception of the bulk density samples, all pretreatment samples of the Off-Site Area One soil were collected from the mixer after blending. This was considered important due to the non-homogeneous nature of this soil.

6.3.2 Post-Treatment Sampling Procedures

Samples of treated waste to be used for testing and analysis were taken from each of the first, second, and third cubic-yard forms immediately after they were filled with the treated waste slurry. These samples were collected by a long-handled scoop and transferred to cylindrical waxed cardboard molds, PVC molds, or other previously designated sample containers. The molds were individually labeled, placed in open storage containers, covered with plastic sheeting, and allowed to cure for 28 days in a heated building (approximately 50 to 70 °F) at the site. Figure 7 shows the samples of treated waste material in the cylindrical molds immediately after collection.

Some of the treated waste slurry was also used for field testing. The results of these tests are reported in Appendix D.

A single one-cubic-yard mold from each of the four batches (reagent mix with sand and three batches of solidified waste) was allowed to cure for the first 24 hours after mixing in a heated (approximately 50 to 70 °F) building. The remaining molds cured at ambient outdoor temperatures (approximately 25 to 40 °F). After one to three days outdoors, all cubic-yard molds were transferred to a non-heated warehouse for the remainder of the curing period.

After the curing period the samples to be used for testing and analyses were removed from the heated building at the site and transported to the analytical laboratory for analyses.

The field demonstration of the Soliditech SITE technology produced 14 large monoliths, initially contained within one-cubic-yard plywood molds. A permanent brass marker, with the block number stamped on it, was imbedded in the top corner of each block before the material had completely hardened. For purposes of describing specific features of each block, this marker is defined as resting on the Northwest corner. The wastes contained within each square mold were:

No. 1	Clean sand plus Soliditech reagent and proprietary additives
No. 2,3,4	Mixture of waste pile filter cake and oily sludge
No. 5,6,7,8,9,10	Waste pile filter cake material
No. 11,12,13,14	Off-Site Area One contaminated soil

The material in Mold Nos. 1, 3, 7, 12 were placed inside a heated warehouse at 50 to 70 °F to cure overnight, since the temperature was slightly below freezing at night during the week of the field demonstration.

After 28 days, the sides of the plywood mold forms were removed from each monolith. Loose fragments were chipped off the edges of each cubical block. The treated waste monolith blocks were then placed in a compact stack for long-term monitoring. Figures 8 and 9 depict the placement of the blocks.

FIGURE 7

SAMPLES OF TREATED WASTE IN CYLINDRICAL MOLDS

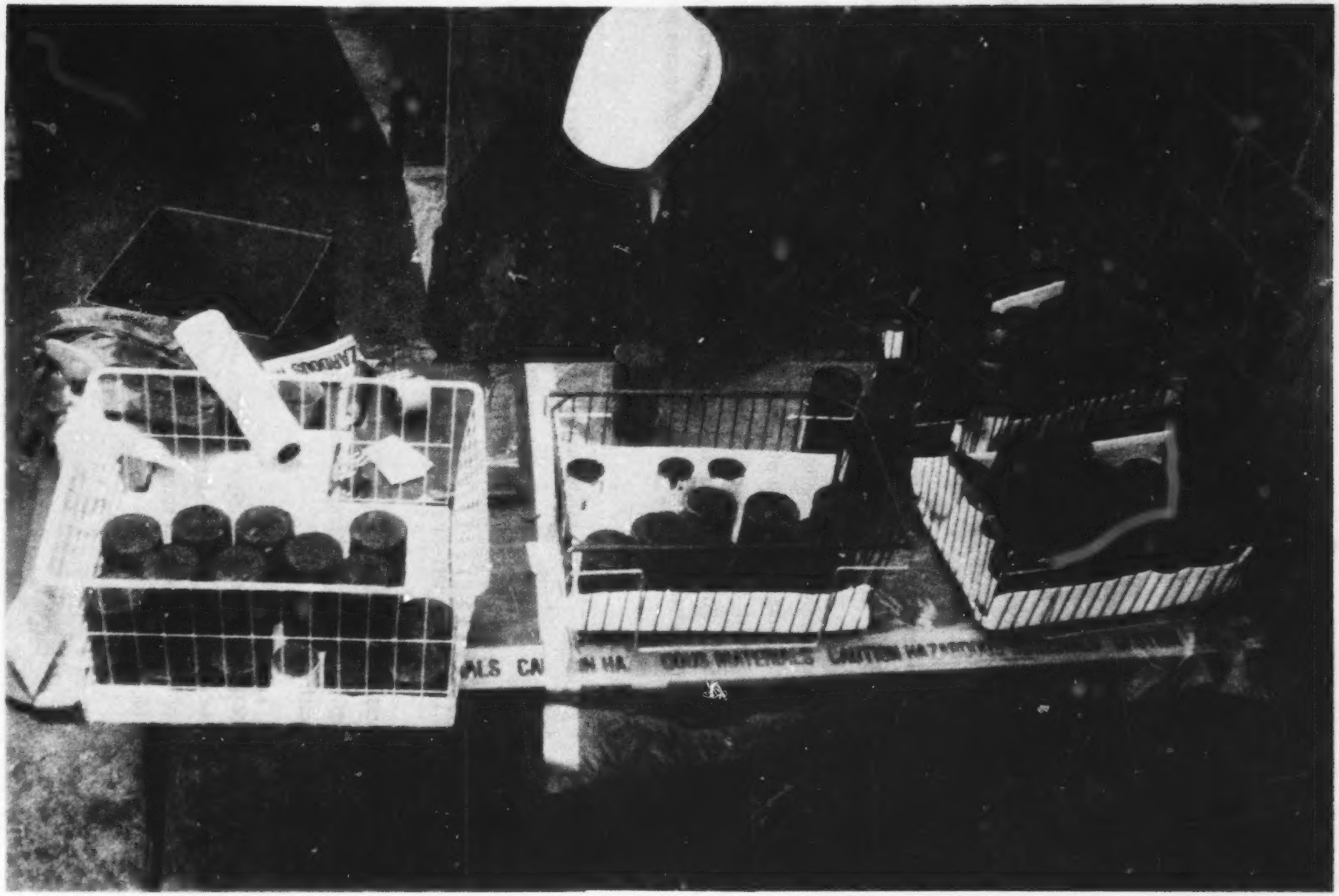


FIGURE 8

LARGE MONOLITHS OF TREATED WASTE BEING PREPARED FOR LONG-TERM STUDY

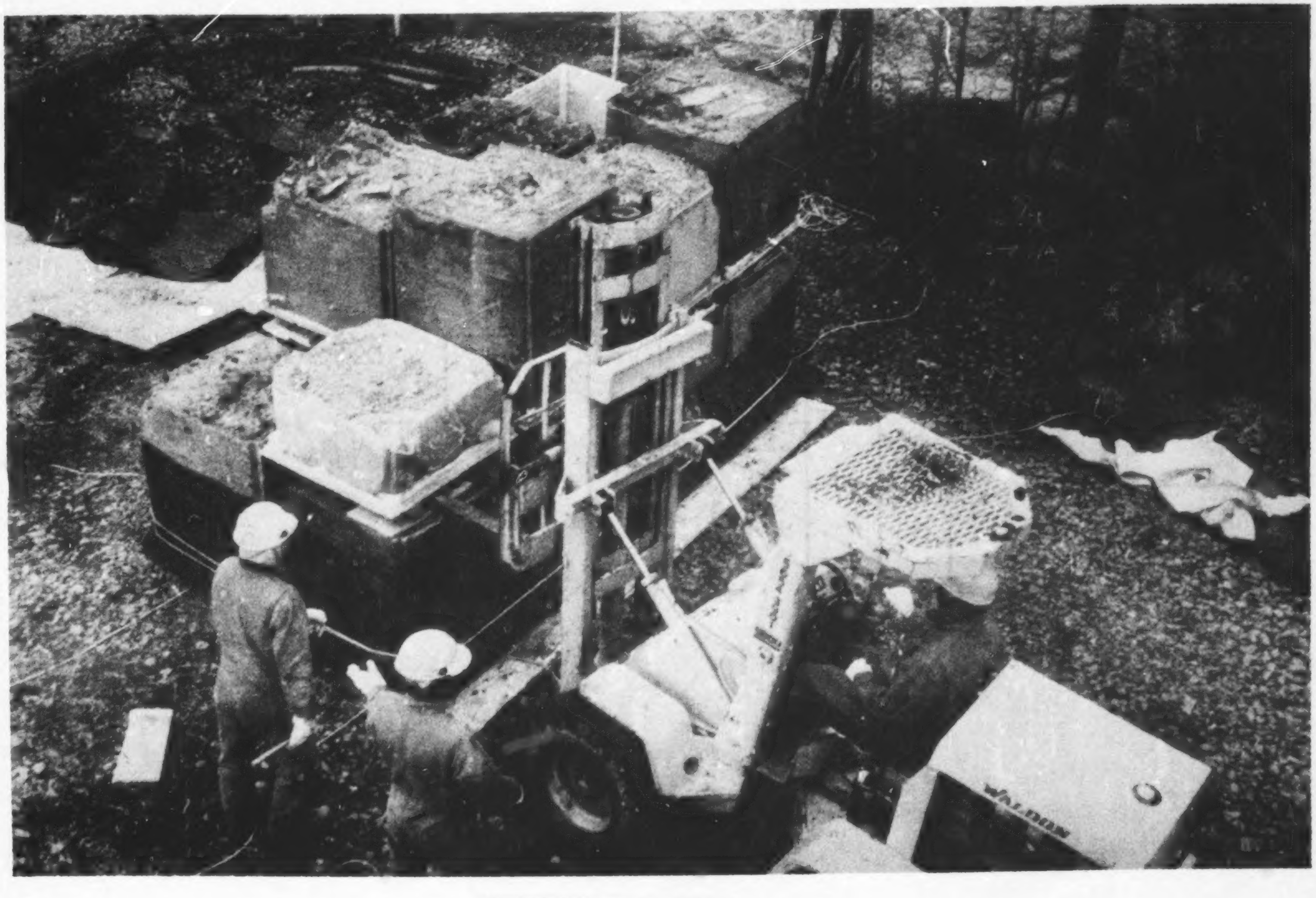


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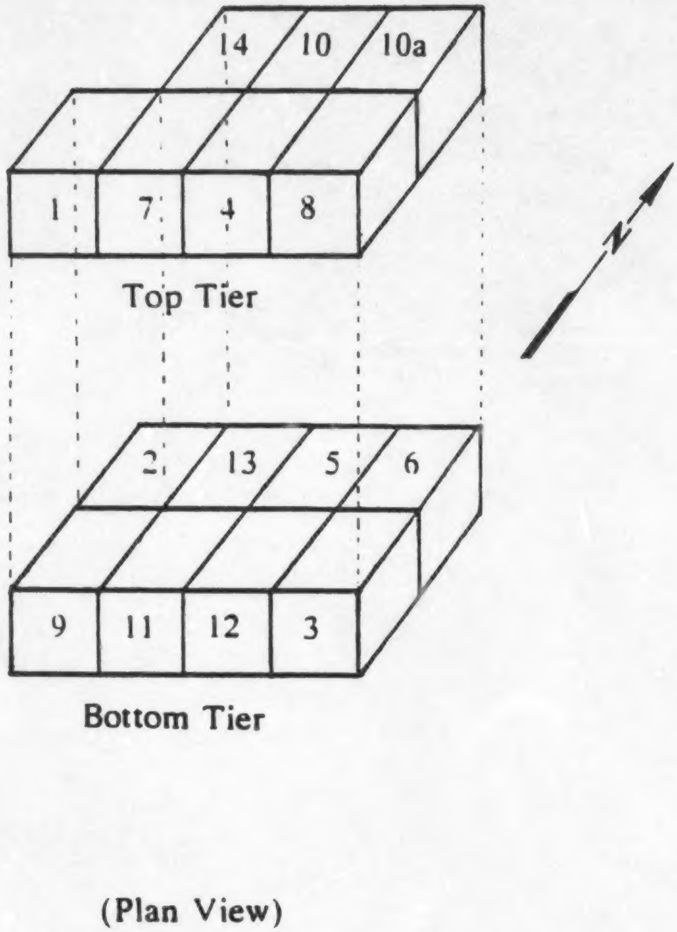
FIGURE 9

STACKING DIAGRAM OF THE TREATED WASTE MONOLITHS



The stack was placed on a double-thickness of 40-mil HDPE, wrapped up the sides of the stack to contain any liquid that might seep from the blocks. The stack was covered with a sheet of 40-mil HDPE to divert precipitation from the blocks. The stack cover was not made airtight; therefore, temperature and humidity influence on the solidified waste material may have some long-term effect.

As the plywood side forms of the treated waste monoliths were removed and the blocks were placed on the stack, initial observations were recorded. Long-term evaluation of the treated waste monoliths will examine:

- Surface spalling
- Grain exfoliation
- Crack and fissure development
- Oxidative discoloration
- Salt efflorescence
- Pore characterization

6.3.3 Sampling Deviations

Pretreatment samples of the filter cake and the Off-Site Area One waste material were, according to the Demonstration Plan, to be taken directly from their native areas. The oily sludge/filter cake material was to be mixed first in the Soliditech mixer and then sampled. These protocols were followed for the filter cake material and the filter cake/oily sludge wastes. However, due to perceived non-homogeneity in the Off-Site Area One soil, this waste was first thoroughly mixed in the Soliditech mixer before it was sampled for chemical analyses, leaching tests, moisture content, and particle size. The samples to determine bulk density were collected from the native area using Shelby tubes, as specified in the Demonstration Plan.

7.0 PERFORMANCE DATA AND EVALUATION

The Soliditech SITE technology evaluation was based on laboratory test results on samples of waste materials collected before and after treatment. Physical tests included bulk density of treated waste, water content, particle size analysis, permeability of the treated waste, unconfined compressive strength, and wet/dry and freeze/thaw tests on treated waste. Chemical tests were applied for pH, Eh, metals, polychlorinated biphenyls, volatile organic compounds, semivolatile organic compounds, loss on ignition, oil and grease, and acid neutralization capacity/neutralization potential. Extraction tests included TCLP extraction, EP toxicity, BET, ANS 16.1, and WILT.

7.1 PHYSICAL TESTS

Physical tests performed on the pretreatment and post-treatment wastes included bulk density, water content, loss on ignition, particle size analysis, permeability, unconfined compressive strength, wet/dry weathering, freeze/thaw weathering, and acid neutralization capacity. These waste characteristics are summarized in Table 14 and are discussed in the following sections. The detailed data are presented in Appendix A, Tables A.1 through A.7 and Figures A.1 through A.3.

7.1.1 Bulk Density

Bulk density of the pretreatment wastes ranged from 1.14 to 1.26 g/cm^3. Waste material from Off-Site Area One was greatest in bulk density because of the high content of indigenous soil. The filter cake waste was lowest in bulk density because this waste contained a high proportion of diatomaceous earth. Bulk densities of the post-treatment wastes were significantly greater than the pretreatment wastes owing to the addition of cement. Bulk densities for the post-treatment samples ranged from 1.43 to 1.68 g/cm^3.

7.1.2 Water Content

Water content of the pretreatment Off-Site Area One and filter cake wastes were 23.5 and 28.7 percent, respectively. After the solidification/stabilization treatment, these wastes contained 12.6 and 21.0 percent water, respectively. The difference in water content after solidification/stabilization was apparently related to the amount of water and cement added in the solidification/stabilization process. The filter cake/oily sludge waste contained 58.1 percent water prior to treatment and 14.7 percent in the post-treatment samples.

TABLE 14

CHEMICAL AND PHYSICAL ANALYSES OF UNTREATED AND TREATED WASTE

	Filter Cake		Filter Cake/Oily Sludge Mixture		Off-Site Area One		Reagent Mix
	Untreated	Treated	Untreated	Treated	Untreated	Treated	
Volatile Organic Compounds (mg/Kg)							
Ethyl Benzene	<1.5	<1.5	4.3	<2.2	<1.5	<1.5	NA
Tetrachloroethene	<1.5	<1.5	1.6	<1.5	<1.5	<1.5	NA
Toluene	<1.5	<1.5	8.4	<4.9	8.3	<7.9	NA
Trichloroethene	<1.5	<1.5	3.3	<2.2	<1.5	<1.5	NA
Xylenes	<1.5	<1.5	32	<18	2.2	<2.2	NA
Semivolatile Organic Compounds (mg/Kg)							
Butyl benzyl phthalate	<10	<5.0	49	<3.3	49	4.3	<1.0
o-Cresol	<10	<5.0	<10	<3.3	<5.0	<3.3	<1.0
p-Cresol	<10	14	<10	4.6	<5.0	<3.3	<1.0
2,4-Dimethylphenol	<10	<5.0	<10	3.7	<5.0	<3.3	<1.0
Bis(2-Ethylhexyl)phthalate	<10	10	<10	<3.3	24	8.2	<1.0
2-Methylnaphthalene	<10	<5.0	14	4.4	6.2	3.8	<1.0
Naphthalene	<10	<5.0	<10	<3.3	<5.0	<3.3	<1.0
Phenol	<10	12	<10	4.8	<5.0	<3.3	<1.0
PCBs (mg/Kg)							
Aroclor-1242	9.3	6.3	16	6.2	29	33	<0.0020
Aroclor-1260	19	10	27	8.4	14	7.5	<0.0040
Metals (AA) (mg/Kg)							
Arsenic	26	28	14	40	94	92	39
Mercury	<0.040	<0.040	<0.040	<0.040	0.16	0.17	<0.040
Selenium	<0.20	<0.20	<0.20	<0.20	0.23	<0.20	<0.20
Thallium	0.17	0.15	0.052	0.12	<0.050	<0.10	0.17
Metals (ICPES) (mg/Kg)							
Aluminum	8,400	17,000	5,500	18,000	4,000	11,000	22,000
Barium	1,900	780	1,600	1,000	700	580	1,700
Beryllium	0.17	<0.10	0.13	0.23	0.23	<0.10	0.54
Cadmium	0.37	0.50	1.0	1.0	1.5	0.70	1.2
Calcium	1,000	110,000	1,200	190,000	4,600	150,000	180,000
Chromium	4.7	20	5.7	28	11	29	38
Copper	21	28	34	43	33	43	60
Lead	2,200	680	2,500	850	650	480	20
Nickel	2.7	11	3.0	16	2.7	13	21
Sodium	83	430	950	1,800	93	480	2,500
Zinc	26	23	150	54	120	95	39
Other Chemical & Physical Tests							
Bulk Density (g/cm^3)	1.14	1.43	1.19	1.68	1.26	1.59	NA
Eh (mv)	370	-31	220	-45	100	-63	-60
Loss on Ignition at 550°C (mg/Kg)	540,000	410,000	700,000	340,000	360,000	340,000	NA
Oil and Grease, infrared (mg/Kg)	170,000	77,000	130,000	60,000	28,000	46,000	NA
Permeability (cm/sec)	NA	4.53×10^{-7}	NA	8.93×10^{-9}	NA	3.41×10^{-8}	NA
Unconfined Compressive Strength (psi)	NA	390	NA	860	NA	680	NA
Particle Size	0.32	NA	0.46	NA	0.42	NA	NA
Water Content (%)	28.7	21.0	58.1	14.7	23.5	12.6	NA
pH (pH units)	3.4	11.8	3.6	12.0	7.9	12.0	12.1
Wet/Dry Weathering (% wt. loss)	NA	<1	NA	<1	NA	<1	NA
Freeze/Thaw Weathering (% wt. loss)	NA	<1	NA	≤1	NA	<1	NA

NA: Not Analyzed

7.1.3 Particle Size Distribution

The mean particle size for the pretreatment wastes ranged from 0.32 to 0.46 mm. This particle size range is representative of a fine textured material, which could be found at many hazardous waste sites. Particle size was not determined on the post-treatment wastes because the treated wastes were monolithic solids.

7.1.4 Permeability

Permeability of the untreated wastes was not determined. Permeabilities of the treated wastes ranged from 8.9×10^{-9} cm/sec for the filter cake/oily sludge to 4.5×10^{-7} cm/sec for the filter cake.

7.1.5 Unconfined Compressive Strength (UCS)

UCS determinations were performed on molded samples of solidified waste according to ASTM D1633. UCS values ranged from 390 psi for the filter cake waste to 860 psi for the filter cake/oily sludge. The USC values were inversely proportional to the permeability of the treated waste and directly proportional to the weight fraction of cement mixed with the wastes. The ratio of water and cement to waste varied for each treatment run due to the nature of the untreated waste materials.

7.1.6 Wet/Dry Weathering Test

Wet/dry weathering tests were performed on molded samples of the post-treatment wastes according to method TMSWC-12. Results are expressed as the cumulative weight loss incurred through 12 wet/dry cycles normalized to a control that is not subjected to the wet/dry cycles. Results for all wastes indicate that less than one percent of the cast cylinder weights were lost over the 12 wet/dry cycles. UCS determinations were performed on the cast cylinders after completion of the wet/dry weathering tests. UCS values ranged from 120 psi for the filter cake waste to 220 psi for the filter cake/oily sludge waste.

7.1.7 Freeze/Thaw Weathering Test

Freeze/thaw weathering tests were performed on molded samples of the post-treatment wastes according to method TMSWC-11. Results were reported in the same manner as the wet/dry weathering tests. Results for all wastes indicate that 1 percent or less of the cast cylinder weights were lost over the 12 freeze/thaw cycles. UCS determinations were performed on cast cylinders after completion of the freeze/thaw weathering test. UCS values ranged from 130 psi for the filter cake waste to 290 for the filter cake/oily sludge waste.

7.1.8 Loss on Ignition

The loss on ignition of the pretreatment and post-treatment wastes were determined by ASTM C114. The loss on ignition for the filter cake samples was 54 percent (by weight) for the pretreatment waste and 41 percent for the post-treatment waste. The filter cake/oily sludge had a loss on ignition of 70 percent in the pretreatment waste (due to the high water content of this waste mixture) and 34 percent in the post-treatment waste. The Off-Site Area One samples had a loss on ignition of 36 percent in the pretreatment waste and 34 percent in the post-treatment waste.

7.1.9 On-Site Tests

Field tests performed during the Soliditech demonstration included the Slump of Portland Cement (ASTM C143) and the Homogeneity of Mixing (a modification of ASTM C136 and C142) tests. The slump results were 4.1 inches for the filter cake/oily sludge mixture, 5.8 inches for the filter cake waste, and 5.4 inches for Off-Site Area One. The slump measurement is the vertical difference between the top of the mold and the top of the slumped waste material. The results are proportional to the amount of cement in each of the treated waste mixtures.

The results of the Homogeneity of Mixing test indicate that the treated waste materials in the treated waste slurries were finely divided. The filter cake/oily sludge mixture had the highest percentage of solids retained on the 0.25 to 0.75 inch sieves, 4.3 percent by weight, while the values for the filter cake waste and Off-Site Area One waste were 2.3 and 1.9 percent by weight, respectively. The results of this test appear to correlate with visual observations of unmixed material in the filter cake oily sludge test run.

The results of these field tests may not be adequate to determine the homogeneity of mixing of treated waste material. The development of quantitative field tests would be desirable.

7.2 CHEMICAL TESTS

Results of the chemical tests performed on the pre- and post-treatment waste samples, as well as the reagent mix, are summarized in Table 14. These tests analyzed the pre- and post-treatment wastes to determine the changes in chemical composition after treatment. Chemical composition is not necessarily an indicator of contaminant mobility. Section 7.3 presents the results of leaching tests. The leaching tests may indicate contaminant mobility. Tables 15 and 16 summarize the results of the chemical tests performed on the sand used for the reagent mix and the residual material cleaned from the mixer prior to its use. The detailed chemical data are presented in Appendix A, Tables A.1 through A.9. Chemical characteristics analyzed include pH, Eh, metals, PCBs, VOCs, SVOCs, loss on ignition, and oil and grease.

7.2.1 pH

The pH of the pre- and post-treatment wastes and the reagent mix samples was determined by U.S. EPA Method 9045. The pH of the pretreatment Off-Site Area One waste was 7.9. The filter cake and filter cake/oily sludge wastes had a pH values of 3.4 and 3.6. The post-treatment wastes and the reagent mix had pH values of 11.8 and 12.1. This extremely high pH reflected the alkalinity associated with the cement in the solidification/ stabilization process.

7.2.2 Eh

The Eh of the pretreatment and post-treatment wastes and the reagent mix samples was determined with an Eh electrode on waste/water slurries prepared for the determination of pH by U.S. EPA Method 9045. The Eh of the pretreatment wastes ranged from 100 millivolts (mv) for Off-Site Area One to 370 mv for the filter cake. The post-treatment wastes and the reagent mix had Eh values ranging from -31 to -63 mv.

7.2.3 Metals

Several U.S. EPA methods were employed to determine the concentration of metals in the predemonstration blank, pretreatment and post-treatment wastes, and the reagent mix. These digestion and analytical methods were discussed in Section 5.6.2.

The pretreatment wastes from the filter cake contained 2,200 mg/Kg lead, 1,900 mg/Kg barium, 26 mg/Kg arsenic, 21 mg/Kg copper, and 26 mg/Kg zinc. Lead and barium concentrations in the post-treatment filter cake waste contained higher concentrations of chromium (20 mg/Kg) and nickel (11 mg/Kg) than the pre-treatment waste (4.7 and 2.7 mg/Kg, respectively). The post-treatment wastes from the filter cake also contained elevated levels of calcium, aluminum, and sodium due to the addition of cement to the wastes. All other metal concentrations were approximately the same in the pre- and post-treatment filter cake wastes.

TABLE 15

CHEMICAL ANALYSIS OF SAND

Metals (AA) (mg/Kg)	
Arsenic	0.11
Mercury	<0.050
Selenium	<0.20
Thallium	<0.20
Metals (ICPES) (mg/Kg)	
Aluminum	110
Barium	<1.0
Beryllium	<0.20
Cadmium	<0.50
Calcium	<100
Chromium	<3.0
Copper	<2.0
Lead	<5.0
Nickel	<2.0
Sodium	<100
Zinc	<2.0

Note: The sand was used as a waste surrogate in the reagent mix test run.

TABLE 16

CHEMICAL ANALYSIS OF PREDEMONSTRATION BLANK

Volatile Organic Compounds (mg/Kg)	
Ethyl benzene	9.4
Tetrachloroethene	17
Toluene	7.1
Trichloroethene	2.2
Xylenes	51
Semivolatile Organic Compounds (mg/Kg)	
Butyl benzyl phthalate	<5.0
o-Cresol	<5.0
p-Cresol	<5.0
2,4-Dimethylphenol	<5.0
Bis(2-ethylhexyl)phthalate	14
2-Methylnaphthalene	150
Naphthalene	55
Phenol	50
PCBs (mg/Kg)	
Arcoclor-1242	<0.99
Arcoclor-1260	<2.0
Metals (AA) (mg/Kg)	
Arsenic	95
Mercury	1.0
Selenium	<0.20
Thallium	0.10
Metals (ICPES) (mg/Kg)	
Aluminum	43,000
Barium	2,500
Beryllium	2.2
Cadmium	7.1
Calcium	130,000
Chromium	780
Copper	380
Lead	160
Nickel	75
Sodium	5,400
Zinc	840

Note: The predemonstration blank consisted of residual material removed from the large Soliditech mixer prior to treatment of waste.

The pretreatment filter cake/oily sludge waste mixture contained 2,500 mg/Kg lead, 1,600 mg/Kg barium, 150 mg/Kg zinc, 14 mg/Kg arsenic, and 34 mg/Kg copper. Lead, barium, and zinc concentrations in the post-treatment filter cake/oily sludge wastes decreased to 850 mg/Kg, 1,000 mg/Kg, and 54 mg/Kg, respectively. The post-treatment filter cake/oily sludge wastes also contained higher concentrations of chromium (28 mg/Kg) and nickel (16 mg/Kg) than in the pretreatment wastes (5.7 mg/Kg and 3.0 mg/Kg, respectively). The post-treatment wastes from the filter cake/oily sludge contained elevated levels of calcium, aluminum, and sodium due to the cement added to the wastes. All other metal concentrations were approximately the same in the pre- and post-treatment wastes.

The pretreatment Off-Site Area One waste contained 650 mg/Kg lead, 700 mg/Kg barium, 120 mg/Kg zinc, 94 mg/Kg arsenic, and 33 mg/Kg copper. Mercury (0.16 mg/Kg) was also found in the Off-Site Area One pretreatment waste. Lead (480 mg/Kg), barium (580 mg/Kg), and zinc (95 mg/Kg) in the Off-Site Area One post-treatment waste also decreased in concentration compared to the pretreatment waste. The post-treatment Off-Site Area One waste also contained higher concentrations of chromium (29 mg/Kg) and nickel (13 mg/Kg) than the pretreatment wastes (11 mg/Kg and 2.7 mg/Kg, respectively). The post-treatment Off-Site Area One wastes also contained elevated levels of calcium, aluminum, and sodium due to the cement added to the wastes. All other metal concentrations were approximately the same in the pre- and post-treatment wastes.

The reagent mix contained large concentrations of calcium (18 percent by weight), aluminum (2.2 percent), and sodium (0.25 percent) as expected. The reagent mix also contained appreciable amounts of the following metals; arsenic (59 mg/Kg), barium (1,700 mg/Kg), chromium (38 mg/Kg), copper 60 mg/Kg), nickel (21 mg/Kg), and zinc (39 mg/Kg). The decreasing trends in metal concentrations in the post-treatment versus pretreatment wastes can be attributed to dilution by the reagent mix when the reagent mix contained a lower concentration of an element than the pretreatment waste (as in the case of lead, barium, and zinc). Where the reagent mix contained a higher concentration of a metal than the pretreatment waste, the concentration of that metal in the post-treatment waste was increased (as with chromium, nickel, aluminum, calcium, and sodium). The concentration of a metal remained relatively constant if the pretreatment waste and the reagent mix contained similar concentrations.

The predemonstration blank (scrapings of the residue from the inside of the Soliditech mixing unit before this demonstration) contained high levels of the elements listed in Table 16. The impact of the predemonstration solids was considered negligible after dilution with contaminated soils and reagent.

A comparative analysis of the pretreatment and post-treatment wastes showed decreasing trends in metal concentrations. Where the reagent mix contained a higher concentration of a metal, the concentration of that metal in the post-treatment waste was increased. The concentration of a metal remained relatively constant if the pretreatment waste and the reagent mix contained similar concentrations.

7.2.4 Polychlorinated Biphenyls (PCBs)

The pretreatment and post-treatment wastes, reagent mix, and predemonstration blank were analyzed for PCBs by U.S. EPA Method 8080. PCBs were not detected in the predemonstration blank or the reagent mix samples.

Aroclor 1242 and Aroclor 1260 (PCBs) were found in the pretreatment and post-treatment wastes. The pretreatment filter cake samples contained 9.0 mg/Kg Aroclor 1242 and 19 mg/Kg Aroclor 1260. The post-treatment filter cake samples contained slightly lower concentrations of these Aroclors (6.3 and 10 mg/Kg, respectively).

The pretreatment filter cake/oily sludge samples contained 16 mg/Kg Aroclor 1242 and 27 mg/Kg Aroclor 1260. The post-treatment filter cake/oily sludge samples contained 6.2 to 8.4 mg/Kg of each of these Aroclors, respectively.

The pretreatment Off-Site Area One samples contained 29 mg/Kg Aroclor 1242 and 14 mg/Kg Aroclor 1260. Post-treatment samples from Off-Site Area One contained 33 mg/Kg of Aroclor 1242 and 7.5 mg/Kg Aroclor 1260.

7.2.5 Volatile Organic Compounds (VOCs)

The pretreatment and post-treatment wastes were analyzed for VOCs by U.S. EPA Method 8240. VOCs were not detected in the pretreatment filter cake samples. The pretreatment filter cake/oily sludge samples contained 32 mg/Kg total xylenes, 4.3 mg/Kg ethyl benzene, 8.4 mg/Kg toluene, and 3.3 mg/Kg trichloroethene. Tetrachloroethylene was detected in one of the pretreatment filter cake/oily sludge samples. Off-Site Area One pretreatment wastes contained 8.3 mg/Kg toluene and 2.2 mg/Kg total xylenes. 1,2-Dichloroethane was detected in one of the pretreatment Off-Site Area One samples. VOCs were not detected in any of the post-treatment waste samples.

The predemonstration blank, which consisted of scrapings of residue from the inside of the Soliditech mixing unit before this demonstration was initiated, was found to contain a number of VOCs. Due to the absence of VOCs in the post-treatment samples, these scrapings were not considered to be a source of VOC contamination in this demonstration.

Organic emissions were monitored during waste collection and treatment with no positive results. It is possible that organics were lost during these operations.

7.2.6 Semivolatile Organic Compounds (SVOCs)

The pretreatment and post-treatment wastes, reagent mix, and predemonstration blank were analyzed for SVOCs by U.S. EPA Method 8270.

SVOCs were not detected in the pretreatment filter cake samples. Phenol (12 mg/Kg) and p-cresol (14 mg/Kg) were found at concentrations near the detection limit in the post-treatment filter cake samples.

The pretreatment filter cake/oily sludge contained 49 mg/Kg of butyl benzyl phthalate and 14 mg/Kg of 2-methylnaphthalene. Naphthalene (9.4 mg/Kg) and o-cresol (5.0 mg/Kg) were detected in one of three pretreatment filter cake/oily sludge samples. The post-treatment filter cake/oily sludge samples contained 2,4-dimethylphenol, 2-methylnaphthalene, phenol, bis(2-ethylhexyl)phthalate, and p-cresol at concentrations less than 5 mg/Kg each.

Off-Site Area One pretreatment wastes contained 6.2 mg/Kg of 2-methylnaphthalene, 40 mg/Kg of butyl benzyl phthalate, and 24 mg/Kg of bis(2-ethylhexyl)phthalate. Di-n-butylphthalate was detected in one of the pretreatment Off-Site Area One samples. The post-treatment Off-Site Area One samples contained detectable quantities of 2-methylnaphthalene, butyl benzyl phthalate, and bis(2-ethylhexyl)phthalate.

SVOCs were not detected in the reagent mix samples.

The predemonstration blank contained a number of SVOCs. These included: 2-methylnaphthalene (150 mg/Kg), anthracene (30 mg/Kg), hexachlorobenzene (370 mg/Kg), naphthalene (55 mg/Kg), pentachlorobenzene (26 mg/Kg), phenanthrene (45 mg/Kg), and phenol (50 mg/Kg). These scrapings were not considered a source of SVOC contamination in this demonstration because concentrations of SVOCs were low and the amount of residue was extremely small compared to the amount of waste being treated.

7.2.7 Oil and Grease

The oil and grease content of the pretreatment and post-treatment wastes was determined by a sonication extraction of the soil (modified U.S. EPA Method 3550) followed by U.S. EPA Method 413.2. The oil and grease content of the filter cake samples was 17 percent (by weight) in the pretreatment waste and 7.7 percent in the post-treatment waste. The filter cake/oily sludge samples contained 13 percent oil and grease in the pretreatment waste and 6.0 percent in the post-treatment waste. Off-Site Area One results indicated that the oil and grease concentrations increased from 2.8 percent in the pretreatment waste to 4.6 percent in the post-treatment waste. The reason for this increase is not clear, but may be related dispersion of the oil and grease by treatment or to the increased pH of the treated waste.

7.2.8 Acid Neutralization Capacity/Neutralization Potential

The acid neutralization capacity results for the pretreatment wastes are presented in Table A.6 in Appendix A. These data indicate that the pretreatment wastes have very low acid neutralization capacity. The pH of Off-Site Area One was reduced from 8.7 to 4.4 with the addition of the first aliquot of acid. The filter cake and filter cake/oily sludge wastes had initial pH values of 3.4 and 3.6 prior to any acid addition.

Acid neutralization results for the post-treatment wastes could not be determined by this method. The pH levels of the post-treatment wastes were all greater than 11.8 and the addition of acid did not appreciably change this pH. The neutralization capacity of the post-treatment soils was determined by "Field and Laboratory Methods Applicable to Overburdens and Minesoils" (U.S. EPA, 1978). The neutralization potential, expressed as percent $CaCO_3$, was 43.3 percent for the reagent mix, 31.3 percent for the filter cake, 43.1 percent for the filter cake/oily sludge, and 39.9 percent for Off-Site Area One.

7.3 LEACHING TESTS

The results of the leaching tests are summarized in Tables 17 through 25. The detailed chemical data is presented in Appendix A, Tables A.10 through A.44, which are attached to this report. A discussion of these results is given below.

7.3.1 TCLP

The Toxicity Characteristic Leaching Procedure (TCLP) was performed on the pretreatment and post-treatment wastes and the reagent mix. This procedure, described in the Federal Register, 1986 (40 CFR, Part 268), is used to determine the extractability of organic and inorganic constituents using an acidic extraction solution to simulate municipal solid waste codisposal. The TCLP leachate data are summarized in Table 17. The detailed data are provided in Appendix A, Tables A.10 through A.13.

7.3.1.1 Reagent Mix

The TCLP leachates produced from the reagent mix samples had pH of 11.4, and Eh of -23 mv. The leachates contained 8,600 mg/L total dissolved solids (TDS). PCBs and oil and grease were not detected in any of the reagent mix TCLP leachates. Barium, calcium, and sodium were found at concentrations of 4.0, 1,900, and 34 mg/L, respectively. Aluminum and chromium were present at detection limit concentrations. Small concentrations (<0.07 μg/L) of six VOCs (acetone, 2-butanone, 4-methyl-2-pentanone, ethyl benzene, toluene, and total xylenes) were present in the TCLP leachates of the reagent mix. SVOCs were not detected in the reagent mix leachates. The strong alkalinity of these samples made it difficult to maintain the proper pH when extracting the acid compounds. Thus, surrogate recoveries and matrix spikes indicate that the acid compound results are biased 50 to 100 percent low.

7.3.1.2 Filter Cake

The pretreatment TCLP leachate samples from the filter cake had a pH of 4.6 and an Eh of 270 mv. The post-treatment leachates had a pH of 10.8 and an Eh of -28 mv. The pretreatment leachates contained 4,500 mg/L TDS while the post-treatment leachates had 8,500 mg/L TDS. Oil and grease was found at 1.4 mg/L in the pretreatment leachate and 4.4 mg/L in the post-treatment leachate. PCBs were not detected in any of the pretreatment or post-treatment TCLP leachates.

The pretreatment leachates contained detectable quantities of arsenic (0.0050 mg/L), lead (4.3 mg/L), zinc (0.28 mg/L), and copper (0.040 mg/L). Arsenic, lead and zinc were not detected, and copper was found at 0.023 mg/L in the post-treatment samples. Chromium was present in low concentrations (0.063 mg/L) in the post-treatment leachates. Calcium concentrations in the post-treatment leachates were greater than in the pretreatment leachates, 9.0 versus 1800 mg/L. Sodium decreased from 1,100 mg/L in the pretreatment leachates to about 13

TABLE 17
CHEMICAL ANALYSES OF TCLP EXTRACT FROM UNTREATED AND TREATED WASTE MATERIALS

	Filter Cake		Filter Cake/Oily Sludge Mixture		Off-Site Area One		
	Untreated	Treated	Untreated	Treated	Untreated	Treated	Reagent Mix
Volatile Organic Compounds (µg/L)							
Acetone	250	<210	1000	<820	200	<160	0.058
Benzene	<2.0	<2.0	8.2	<4.6	<2.0	<2.0	<0.0020
2-Butanone	<10	<10	29	<10	<10	<10	0.011
Ethyl benzene	<2.0	<2.0	8.9	<3.7	4.7	<3.0	<0.0020
4-Methyl-2-pentanone	4.3	<2.0	60	<55	370	<8.4	0.069
Methylene chloride	13	<10	21	<18	<10	<10	<0.010
Tetrachloroethene	<2.0	<2.0	2.9	<2.0	<2.0	<2.0	<0.0020
Toluene	<2.0	<2.0	55	<24	270	<110	0.047
1,1,1-Trichloroethane	4.0	<2.0	<2.0	<2.0	<2.0	<2.0	<0.0020
Trichloroethene	<2.0	<2.0	27	<13	<2.0	<2.0	<0.0020
Xylenes	<2.0	<2.0	57	<14	26	<8.2	0.012
Semivolatile Organic Compounds (µg/L)							
Benzyl alcohol	<10	<10	<10	<10	57	72	<10
Butyl benzyl phthalate	<10	<10	<10	<10	36	<10	<10
o-Cresol	<10	62	44	88	18	13	<10
p-Cresol	<10	440	47	340	<10	110	<10
2,4-Dimethylphenol	<10	20	93	130	10	26	<10
Phenol	<10	630	200	340	<10	100	<10
PCBs (ug/L)							
Arcoclor-1242	<0.42	<0.45	<0.43	<0.11	<0.42	<0.44	<0.11
Arcoclor-1260	<0.84	<0.90	<0.86	<0.21	<0.84	<0.87	<0.22
Metals (AA) (mg/L)							
Arsenic	0.0050	<0.0020	0.014	<0.0020	0.19	0.017	<0.0020
Lead	NA	0.0020	NA	0.014	0.55	0.012	NA
Metals (ICPES) (mg/L)							
Aluminum	0.50	<0.20	0.28	0.47	0.60	0.40	0.77
Barium	1.4	1.3	2.5	5.1	1.6	2.3	4.0
Cadmium	0.0052	<0.0050	0.0093	<0.0050	<0.0050	<0.0050	<0.0050
Calcium	9.0	1,800	21	1,900	190	1,900	1,900
Chromium	<0.030	0.063	<0.030	<0.030	<0.030	0.040	0.063
Copper	0.040	0.023	0.023	<0.020	<0.020	0.037	<0.020
Lead	4.3	<0.20	5.4	<0.050	0.46	<0.050	<0.050
Nickel	<0.020	<0.020	0.027	<0.020	0.033	<0.020	<0.020
Sodium	1,100	13	1,200	43	1,200	16	34
Zinc	0.28	<0.020	1.3	<0.020	0.63	<0.020	<0.020
Other Chemical Tests							
Eh (mv)	270	-28	210	-35	190	-57	-23
Filterable Residue (TDS) (mg/L)	4,500	8,500	5,200	8,600	6,300	9,000	8,600
Oil & Grease, infrared (mg/L)	1.4	4.4	1.6	2.4	1.9	12	<0.40
pH (pH units)	4.6	10.8	4.8	11.6	5.1	11.5	11.4

NA: Not Analyzed

mg/L in the post-treatment leachates. The different sodium levels are a result of the different extraction fluids recommended by the TCLP procedure for solids with different pHs. The extraction fluid for the pretreatment leachates contained sodium hydroxide, while the extraction fluid used for the post-treatment leachates did not.

Three VOCs -- acetone, 4-methyl-2-pentanone, and methylene chloride -- were found in the zero head-space TCLP leachates of the filter cake. These compounds were found at similar levels in the laboratory blank and are therefore considered to be laboratory contaminants. VOCs were not detected in the post-treatment leachates.

SVOCs were not detected in the pretreatment TCLP leachates of the filter cake area. Post-treatment leachates contained phenol (630 μg/L), p-cresol (440 μg/L), o-cresol (62 μg/L), and 2,4-dimethylphenol (20 μg/L).

7.3.1.3 Filter Cake/Oily Sludge

The pretreatment TCLP leachate samples from the filter cake/oily sludge had a pH of 4.8 and an Eh of 210 mv. The post-treatment leachates had a pH of 11.6 and an Eh of -35 mv. The pretreatment leachates contained 5,200 mg/L TDS; the post-treatment leachates 8,600 mg/L TDS. Oil and grease was found at 1.6 mg/L in the pretreatment leachate and 2.4 mg/L in the post-treatment leachate. PCBs were not detected in any of the pretreatment or post-treatment TCLP leachates.

The pretreatment leachates contained low concentrations of arsenic (0.014 mg/L), lead (5.4 mg/L), and zinc (1.3 mg/L). Arsenic, lead and zinc were not detected in the post-treatment leachates. Barium and calcium concentrations were lower in the pretreatment leachates than in the post-treatment leachates, 2.50 versus 5.1 mg/L and 21 versus 1,900 mg/L, respectively. Sodium decreased from 1,200 mg/L in the pretreatment leachates to 43 mg/L in the post-treatment leachates. The different sodium levels are a result of the different extraction fluids recommended by the TCLP procedure for solids with different pHs. The extraction fluid for the pretreatment leachates contained sodium hydroxide, while the extraction fluid used for the post-treatment leachates did not.

Acetone (1,000 μg/L), 4-methyl-2-pentanone (60 μg/L), methylene chloride (21 μg/L), total xylenes (57 μg/L), trichloroethene (27 μg/L), and toluene (55 μg/L) were found in the pretreatment zero head-space TCLP leachates of the filter cake/oily sludge for VOCs.

Tetrachloroethylene, ethyl benzene, benzene, and 2-butanone were also present at detection limit levels in the pretreatment leachates. The acetone, 4-methyl-2-pentanone, and methylene chloride were found at similar levels in the laboratory blank and are therefore considered to be laboratory contaminants for the pretreatment leachates. VOCs were not detected in the post-treatment leachates.

Four SVOCs were found in the pretreatment TCLP leachates of the filter cake/oily sludge area. These were 2,4-dimethylphenol (93 μg/L), phenol (200 μg/L), p-cresol (47 μg/L), and o-cresol (44 μg/L). Post-treatment leachates contained larger concentrations of these compounds: 2,4-dimethylphenol (130 μg/L), phenol (340 μg/L), p-cresol (340 μg/L), and o-cresol (88 μg/L).

7.3.1.4 Off-Site Area One

The pretreatment TCLP leachate samples from the Off-Site Area One had a pH of 5.1 and an Eh of 190 mv. The post-treatment leachates had a pH of 11.5 and an Eh of -57 mv. The pretreatment leachates contained 6,300 mg/L TDS; the post-treatment leachates 9,000 mg/L TDS. Oil and grease was found at 1.9 mg/L in the pretreatment leachate and 12 mg/L in the post-treatment leachate. PCBs were not detected in any of the pretreatment or post-treatment TCLP leachates.

The pretreatment leachates contained low concentrations of arsenic (0.19 mg/L), lead (0.46 mg/L), and zinc (0.63 mg/L). The lead and zinc were not detected in the post-treatment leachates. The arsenic concentration was 0.017 mg/L in the post-treatment leachates. Barium and calcium concentrations were lower in the pretreatment leachates than in the post-treatment leachates, 1.6 versus 2.3 mg/L and 190 versus 1,900 mg/L, respectively. Sodium decreased from 1200 mg/L in the pretreatment leachates to about 16 mg/L in the post-treatment leachates. The different sodium levels are a result of the different extraction fluids recommended by the TCLP procedure for solids with different pHs. The extraction fluid for the pretreatment leachates contained sodium acetate, while the extraction fluid used for the post-treatment leachates contained acetic acid.

Acetone (200 μg/L), 4-methyl-2-pentanone (370 μg/L), total xylenes (26 μg/L), and toluene (270 μg/L) were found in the pretreatment zero head-space TCLP leachates for VOCs. Ethyl benzene was also present at detection limit levels in the pretreatment leachates. The acetone and 4-methyl-2-pentanone were found at similar levels in the laboratory blank and are therefore considered to be laboratory contaminants for the pretreatment leachates. VOCs were not detected in the post-treatment leachates.

Four SVOCs were found in the pretreatment TCLP leachates of the Off-Site Area One area. These were 2,4-dimethylphenol (10 µg/L), benzyl alcohol (57 µg/L), butyl benzyl phthalate (36 µg/L), and o-cresol (18 µg/L). Post-treatment leachates contained increased concentrations of 2,4-dimethylphenol (26 µg/L), benzyl alcohol (72 µg/L), phenol (100 µg/L), and p-cresol (110 µg/L).

7.3.2 EP Toxicity

The Extraction Procedure Toxicity Test (EP) is described in U.S. EPA Method 1310. The leaching test was performed on the pretreatment and post-treatment wastes and the reagent mix. This procedure is also used to determine extractability of organic and inorganic constituents using an acidic extraction solution to simulate municipal solid waste codisposal. The EP is currently used for land disposal restriction regulations. The EP leachate data are summarized in Table 18. The detailed data are presented in Appendix A, Tables A.14 through A.17.

7.3.2.1 Reagent Mix

The EP leachates produced from the reagent mix samples had a pH of 11.3, and Eh of 9.0 mv. The leachates contained 8,700 mg/L total dissolved solids. PCBs and oil and grease were not detected in any of the reagent mix EP leachates. Barium, calcium, and sodium were found at concentrations of 4.3 mg/L, 1,900 mg/L, and 35 mg/L , respectively. Arsenic, selenium, aluminum, and chromium were detected in at least one of the leachates at detection limit concentrations.

7.3.2.2 Filter Cake

The pretreatment EP leachate samples from the filter cake had a pH of 3.8 and an Eh of 320 mv. The post-treatment leachates had a pH of 10.9 and an Eh of -2.0 mv. The pretreatment leachates contained 90 mg/L TDS, and the post-treatment leachates contained 9,500 mg/L TDS. Oil and grease was not detected in the pretreatment leachates. The post-treatment EP leachates contained 4.0 mg/L oil and grease. PCBs were not detected in any of the pretreatment or post-treatment EP leachates.

The pretreatment leachates contained detectable quantities of arsenic (0.010 mg/L), lead (0.25 mg/L), and zinc (0.032 mg/L). The lead and zinc were not detected, and arsenic was found at 0.0023 mg/L in the post-treatment samples. Barium, calcium, and sodium concentrations in the post-treatment leachates were greater than in the pretreatment leachates. Barium increased from 0.21 to 1.4 mg/L, calcium from 4.8 to 2,000 mg/L, and sodium from 1.4 to 15 mg/L. These

TABLE 18

CHEMICAL ANALYSES OF EP EXTRACT FROM UNTREATED AND TREATED WASTE

	Filter Cake		Filter Cake/Oily Sludge Mixture		Off-Site Area One		
	Untreated	Treated	Untreated	Treated	Untreated	Treated	Reagent Mix
PCBs (µg/L)							
Aroclor-1242	<0.43	<0.41	<0.43	<0.42	<0.45	<0.21	<0.020
Aroclor-1260	<0.86	<0.82	<0.86	<0.84	<0.90	<0.42	<0.040
Metals (AA) (mg/L)							
Arsenic	0.010	0.0023	0.011	0.0020	0.18	0.028	<0.0020
Lead	0.26	0.0023	0.55	0.015	0.12	0.012	<0.0020
Mercury	<0.00020	<0.00020	<0.00020	<0.00020	<0.00020	<0.00030	<0.00020
Selenium	<0.0040	<0.0040	<0.0040	<0.0050	<0.0040	<0.0050	0.017
Thallium	<0.0010	<0.0010	0.0013	<0.0010	<0.0010	<0.0010	<0.0010
Metals (ICPES) (mg/L)							
Aluminum	<0.20	<0.20	<0.20	<0.20	0.40	0.20	0.50
Barium	0.21	1.4	1.1	5.7	0.58	2.4	4.3
Beryllium	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cadmium	<0.0050	<0.0050	0.0082	<0.0050	0.0052	<0.0050	<0.0050
Calcium	4.8	2,000	11	2,100	140	2,100	1,900
Chromium	<0.030	0.083	<0.030	0.037	<0.030	<0.030	0.067
Copper	<0.020	0.037	<0.020	<0.020	<0.020	0.060	<0.020
Lead	0.25	<0.050	0.52	<0.050	0.067	<0.050	<0.050
Nickel	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Sodium	1.4	15	58	45	2.1	16	35
Zinc	0.032	<0.020	0.86	<0.020	0.26	<0.020	<0.020
Other Chemical Tests							
Eh (mV)	320	-2.0	220	-30	130	-10	9.0
Filterable Residue (TDS) (mg/L)	90	9,500	330	9,100	790	9,400	8,700
Oil & Grease, infrared (mg/L)	<0.40	4.0	<0.40	3.1	2.6	11	<0.40
pH (pH units)	3.8	10.9	4.8	11.8	4.8	11.7	11.3

increases can be attributed to portland cement and other proprietary additives used during treatment.

7.3.2.3 Filter Cake/Oily Sludge

The pretreatment EP leachate samples from the filter cake/oily sludge had a pH of 4.8 and an Eh of 220 mv. The post-treatment leachates had a pH of 11.8 and an Eh of -30 mv. The pretreatment leachates contained 330 mg/L of TDS while the post-treatment leachates contained 9,100 mg/L of TDS. Oil and grease was not detected in the pretreatment leachates. The post-treatment EP leachates contained 3.1 mg/L of oil and grease. PCBs were not detected in any of the pretreatment or post-treatment EP leachates.

The pretreatment leachates contained detectable quantities of arsenic (0.011 mg/L), lead (0.52 mg/L), and zinc (0.86 mg/L). Lead and zinc were not detected and arsenic was found at 0.0020 mg/L in the post-treatment samples. Barium and calcium concentrations in the post-treatment leachates were greater than in the pretreatment leachates. Barium increased from 1.1 to 5.7 mg/L and calcium from 11 to 2,100 mg/L. These increases can be attributed to the portland cement and proprietary additives used during treatment.

7.3.2.4 Off-Site Area One

The pretreatment EP leachate samples from the Off-Site Area One had a pH of 4.8 and an Eh of 130 mv. The post-treatment leachates had a pH of 11.7 and an Eh of -10.0 mv. The pretreatment leachates contained 790 mg/L of TDS and the post-treatment leachates contained 9,400 mg/L of TDS. The pretreatment leachates contained 2.6 mg/L of oil and grease. The oil and grease increased to 11 mg/L in the post-treatment EP leachates. PCBs were not detected in any of the pretreatment or post-treatment EP leachates.

The pretreatment leachates contained arsenic (0.18 mg/L), lead (0.067 mg/L), and zinc (0.26 mg/L). Lead and zinc were not detected and arsenic was found at 0.028 mg/L in the post-treatment samples. Barium, calcium, and sodium concentrations in the post-treatment leachates were greater than in the pretreatment leachates. Barium increased from 0.58 to 2.4 mg/L, calcium from 140 to 2,100 mg/L, and sodium from 2.1 to 16 mg/L. These increases can be attributed to portland cement and other proprietary additives used during treatment.

7.3.3 BET

The BET was performed on the pretreatment and post-treatment wastes and on the reagent mix samples. The procedure is described in the modification to the Equilibrium Leaching in the Test Methods for Solidified Waste Characterization - Method 6 (Environment Canada/U.S. EPA, no date). The leach test assesses the leachability of constituents from the waste in deionized water with varying solid-to-liquid ratios. Three solid-to-liquid (S/L) ratios were used for this test: 1:4, 1:20, and 1:100. Leachability of pollutant solutes at all S/L ratios was considered to be insignificant. Leachability data for aluminum, barium, calcium, and sodium at various S/L ratios are being evaluated in parallel with data rom other extraction and leaching tests in order to verify the applicability of the BET to these types of sample materials. Due to the large number of nondetected values in the data, no interpretation of the effects of these solid-to-liquid ratios on chemical leachability were made. The BET leachate data are summarized in Tables 19 through 22. The detailed data are presented given in Appendix A, Tables A.18 through A.29.

7.3.3.1 Reagent Mix

The pH of the three BET leachates ranged from 11.8 for the 1:100 S/L leachate to 12.0 for the 1:20 and 1:4 S/L leachates. The Eh of the leachates ranged from -69 to -80 mv. The TDS and total organic carbon (TOC) concentrations increased from 620 mg/L TDS and 3.0 mg/L TOC in the 1:100 S/L leachate to 2,900 mg/L TDS and 36 mg/L TOC in the 1:4 S/L leachate. PCBs and oil and grease were not detected in any of the reagent mix BET leachates.

Arsenic was found at concentrations of 0.0030, 0.0037, and 0.0073 mg/L in the 1:4, 1:20, and 1:100 S/L leachates. Lead and zinc were not detected in any of the reagent mix leachates. The concentration of barium and sodium increased as the S/L ratio decreased. Barium concentrations increased from 1.6 mg/L in the 1:100 S/L leachates to 27 mg/L in the 1:4 leachates. Sodium concentrations increased from 9.0 mg/L in the 1:100 S/L leachates to 160 mg/L in the 1:4 S/L leachates. Conversely, the S/L concentration of aluminum decreased from 4.8 mg/L in the 1:100 S/L leachate to 0.37 mg/L in the 1:4 S/L leachate. Calcium concentrations were equal, at 540 and 560 mg/L, in the 1:4 and 1:20 S/L leachates, and 210 mg/L in the 1:100 S/L leachate.

TABLE 19

CHEMICAL ANALYSIS OF BET EXTRACT FROM UNTREATED AND TREATED FILTER CAKE WASTE

	Solid-to-Liquid Ratio					
	1:4		1:20		1:100	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
PCBs (μg/L)						
Aroclor-1242	<0.42	<0.43	<0.41	<0.41	<0.42	<0.21
Aroclor-1260	<0.84	<0.86	<0.82	<0.82	<0.84	<0.42
Metals (AA) (mg/L)						
Arsenic	0.072	0.011	0.014	0.0037	0.020	0.0020
Mercury	<0.00020	<0.00020	<0.00020	<0.00030	<0.00020	<0.00020
Selenium	<0.0050	<0.0040	<0.0050	<0.0040	<0.0050	<0.0040
Thallium	<0.0020	<0.0010	<0.0020	<0.0010	<0.0020	<0.0010
Metals (ICPES) (mg/L)						
Aluminum	1.9	0.20	0.23	0.47	0.37	1.1
Barium	0.14	6.3	0.28	3.4	0.47	0.92
Beryllium	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cadmium	0.0073	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
Calcium	30	850	7.3	480	1.2	230
Chromium	<0.030	0.046	<0.030	0.037	<0.030	<0.030
Copper	0.050	0.063	<0.020	0.027	<0.020	<0.020
Lead	0.87	<0.050	0.42	<0.050	0.18	<0.050
Nickel	0.063	<0.020	<0.020	<0.020	<0.020	<0.020
Sodium	2.3	84	<1.0	19	<1.0	4.3
Zinc	0.27	<0.020	0.047	<0.020	0.020	<0.020
Other Chemical Tests						
Eh (mV)	270	-82	290	-92	270	-82
Filterable Residue (TDS) (mg/L)	440	3,800	120	1,700	40	760
Oil & Grease, infrared (mg/L)	0.65	6.3	0.53	2.7	<0.40	<0.40
pH (pH units)	3.7	11.7	3.5	11.7	3.9	11.5
Total Organic Carbon (mg/L)	91	140	28	43	11	14

TABLE 20

CHEMICAL ANALYSIS OF BET EXTRACT FROM UNTREATED AND TREATED FILTER CAKE/OILY SLUDGE MIXTURE

	Solid-to-Liquid Ratio					
	1:4		1:20		1:100	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
PCBs (μg/L)						
Aroclor-1242	<1.1	<0.42	<0.44	<0.42	<0.41	<0.22
Aroclor-1260	<2.2	<0.84	<0.88	<0.84	<0.82	<0.44
Metals (AA) (mg/L)						
Arsenic	0.042	0.0080	0.035	0.0023	0.0083	0.0030
Mercury	<0.00020	<0.00020	<0.00020	<0.00030	<0.00020	<0.00020
Selenium	<0.0050	<0.0040	<0.0050	<0.0040	<0.0050	<0.0040
Thallium	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Metals (ICPES) (mg/L)						
Aluminum	0.36	0.23	<0.20	0.43	<0.20	1.3
Barium	0.83	17	0.78	9.6	0.48	2.6
Beryllium	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cadmium	0.036	<0.0050	0.0062	<0.0050	<0.0050	<0.0050
Calcium	44	730	9.1	750	2.1	440
Chromium	<0.030	<0.030	<0.030	<0.030	<0.030	<0.030
Copper	<0.020	0.030	<0.020	0.023	<0.020	<0.020
Lead	1.7	<0.050	0.43	<0.050	0.14	<0.050
Nickel	0.049	0.023	0.028	<0.020	0.022	<0.020
Sodium	230	250	80	58	17	13
Zinc	2.7	<0.020	0.69	<0.020	0.16	<0.020
Other Chemical Tests						
Eh (mV)	240	-101	220	-99	220	-93
Filterable Residue (TDS) (mg/L)	1,800	3,500	470	2,300	110	1,200
Oil & Grease, infrared (mg/L)	3.2	4.9	2.2	1.3	1.3	0.43
pH (pH units)	3.7	12.0	4.2	11.9	4.4	11.8
Total Organic Carbon (mg/L)	200	110	60	32	21	8.0

TABLE 21

CHEMICAL ANALYSIS OF BET EXTRACT FROM UNTREATED AND TREATED OFF-SITE AREA ONE WASTE

	Solid-to-Liquid Ratio					
	1:4		1:20		1:100	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
PCBs (μg/L)						
Aroclor-1242	<1.1	<0.43	<1.1	<0.21	<0.43	<0.10
Aroclor-1260	<2.3	<0.86	<2.2	<0.42	<0.86	<0.20
Metals (AA) (mg/L)						
Arsenic	0.38	0.067	0.29	0.022	0.19	0.0097
Mercury	<0.00020	<0.00020	<0.00020	<0.00030	<0.00020	<0.00020
Selenium	<0.0040	0.0070	<0.0040	0.0060	<0.0040	<0.0040
Thallium	<0.0010	<0.0020	<0.0010	<0.0020	<0.0010	<0.0020
Metals (ICPES) (mg/L)						
Aluminum	<0.20	<0.20	<0.20	<0.20	0.69	0.83
Barium	0.11	9.7	0.047	5.5	0.023	1.4
Beryllium	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cadmium	0.0068	<0.0050	0.0055	<0.0050	<0.0050	<0.0050
Calcium	150	1,000	58	860	19	410
Chromium	<0.030	<0.030	<0.030	<0.030	<0.030	<0.030
Copper	<0.020	0.17	<0.020	0.057	<0.020	0.020
Lead	<0.050	<0.050	<0.050	0.090	<0.050	<0.050
Nickel	<0.020	0.033	<0.020	<0.020	<0.020	<0.020
Sodium	5.0	80	2.2	19	1.1	4.0
Zinc	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Other Chemical Tests						
Eh (mV)	110	-77	150	-78	100	-50
Filterable Residue (TDS) (mg/L)	1,100	4,600	390	2,600	330	980
Oil & Grease, infrared (mg/L)	16	26	12	15	4.4	3.7
pH (pH units)	8.3	12.1	8.6	12.1	9.0	11.8
Total Organic Carbon (mg/L)	190	120	73	54	30	14

TABLE 22

CHEMICAL ANALYSES OF BET EXTRACT FROM REAGENT MIX

	Solid-to-Liquid Ratio		
	1:4	1:20	1:100
PCBs (μg/L)			
Aroclor-1242	<0.11	<0.11	<0.11
Aroclor-1260	<0.22	<0.22	<0.22
Metals (AA) (mg/L)			
Arsenic	0.0030	0.0037	0.0073
Mercury	<0.00020	<0.00020	<0.00020
Selenium	<0.0020	<0.0020	<0.0020
Thallium	<0.0020	<0.0020	<0.0020
Metals (ICPES) (mg/L)			
Aluminum	0.37	1.8	4.8
Barium	27	10	1.6
Beryllium	<0.0020	<0.0020	<0.0020
Cadmium	<0.0050	<0.0050	<0.0050
Calcium	540	560	210
Chromium	<0.030	<0.030	<0.030
Copper	<0.020	<0.020	<0.020
Lead	<0.050	<0.050	<0.050
Nickel	<0.020	<0.020	<0.020
Sodium	160	39	9.0
Zinc	<0.020	<0.020	<0.020
Other Chemical Tests			
Eh (mV)	-69	-80	-71
Filterable Residue (TDS)(mg/L)	2,900	1,700	620
Oil & Grease, infrared (mg/L)	<0.50	<0.50	<0.40
pH (pH units)	12.0	12.0	11.8
Total Organic Carbon (mg/L)	36	9.7	3.0

7.3.3.2 Filter Cake

The pH of the pretreatment BET leachates ranged from 3.5 to 3.9. The Eh of the pretreatment leachates were about 270 to 290 mv for the leachates of all three S/L ratios. The TDS in the pretreatment leachates increased from 40 mg/L in the 1:100 S/L leachates to 440 mg/L in the 1:4 S/L leachates. The TOC concentrations in the pretreatment leachates also increased, from 11 to 91 mg/L, as the S/L ratio increased. Oil and grease was detected at 0.53 to 0.65 mg/L in the 1:20 and 1:4 S/L leachates, respectively. No oil and grease was detected in the 1:100 S/L leachate. PCBs were not detected in any of the pretreatment leachates.

Ten metals were observed in the pretreatment leachates. Most of these increased in concentration as the S/L ratios increased from 1:100 to 1:4. Metals exhibiting this trend were arsenic (0.020 to 0.072 mg/L), lead (0.18 to 0.87 mg.L), zinc (0.020 to 0.27 mg/L), aluminum (0.37 to 1.9 mg/L), calcium (1.2 to 30 mg/L), and nickel (<0.020 to 0.063 mg/L). Cadmium, copper, nickel, and sodium were also detected in the 1:4 S/L leachate. Barium decreased from 0.47 mg/L in the 1:100 leachate to 0.14 mg/L in the 1:4 leachate.

All of the leachates from the post-treatment filter cake wastes had pH levels between 11.5 and 11.7 and Eh levels of -82 to -92 mv. The TDS in the post-treatment leachates increased from 760 mg/L in the 1:100 S/L leachates to 3830 mg/L in the 1:4 S/L leachates. The TOC concentrations in the post-treatment leachates also increased, from 14 to 140 mg/L, as the solid-to-liquid ratio increased. Oil and grease was detected at 2.7 to 6.3 mg/L in the 1:20 and 1:4 S/L leachates. PCBs were not detected in any of the post-treatment leachates.

Seven metals were observed in the post-treatment leachates. Most of these increased in concentration as the solid-to-liquid ratios increased from 1:100 to 1:4. Metals exhibiting this trend were arsenic (0.0020 to 0.011 mg/L), barium (0.92 to 6.3 mg/L), chromium (<0.030 to 0.046 mg/L), copper (<0.020 to 0.063 mg/L), calcium (230 to 850 mg/L), and sodium (4.3 to 84 mg/L). Aluminum decreased from 1.1 mg/L in the 1:100 S/L leachate to 0.20 mg/L in the 1:4 S/L leachate. Lead, cadmium, nickel, and zinc were not detected in the post-treatment leachates.

7.3.3.3 Filter Cake/Oily Sludge

The pH of the pretreatment BET leachates ranged from 3.7 to 4.4. The Eh of the pretreatment leachates ranged from 220 to 240 mv for the leachates of all three solid-to-liquid ratios. The TDS in the pretreatment leachates increased from 110 mg/L in the 1:100 S/L leachates to 1,800 mg/L in the 1:4 S/L leachates. The TOC concentrations in the pretreatment leachates also increased, from 21 to 200 mg/L, as the solid-to-liquid ratio increased. Oil and grease increased from 1.3 mg/L in the 1:100 leachates to 3.2 mg/L in the 1:4 S/L leachates. PCBs were not detected in any of the pretreatment leachates.

Nine metals were observed in the pretreatment leachates. Most of these increased in concentration as the solid-to-liquid ratios increased from 1:100 to 1:4. Metals exhibiting this trend were arsenic (0.0083 to 0.042 mg/L), barium (0.48 to 0.83 mg/L), lead (0.14 to 1.7 mg/L), zinc (0.16 to 2.7 mg/L), calcium (2.1 to 44 mg/L), and cadmium (<0.0050 to 0.036 mg/L). Aluminum, nickel and copper were also detected in the leachates.

The leachates from the post-treatment filter cake/oily sludge wastes had pH values ranging from 11.9 to 12.0 and Ehs of -93 to -101 mv. The TDS in the post-treatment leachates increased from 1200 mg/L in the 1:100 S/L leachates to 3,500 mg/L in the 1:4 S/L leachates. The TOC concentrations in the post-treatment leachates also increased, from 8.0 to 110 mg/L, as the solid-to-liquid ratio increased. Oil and grease concentrations increased from 0.43 mg/L in the 1:100 leachates to 4.9 mg/L in the 1:4 leachates. PCBs were not detected in any of the post-treatment leachates.

Seven metals were observed in the post-treatment leachates. Most of these increased in concentration as the solid-to-liquid ratios increased from 1:100 to 1:4. Metals exhibiting this trend were arsenic (0.0030 to 0.0080 mg/L), barium (2.6 to 17 mg/L), calcium (440 to 730 mg/L), and sodium (13 to 250 mg/L). Aluminum decreased from 1.3 mg/L in the 1:100 leachates to 0.23 mg/L in the 1:4 leachate. Copper, which was not detected in the pretreatment samples, and nickel were detected in the 1:4 leachates. Cadmium, lead, and zinc were not detected in the post-treatment leachates.

7.3.3.4 Off-Site Area One

The pH of the pretreatment BET leachates ranged from 8.3 to 9.0. The Eh of the pretreatment leachates ranged from 100 to 150 mv. The TDS in the pretreatment leachates increased from 330 mg/L in the 1:100 S/L leachates to 1,100 mg/L in the 1:4 S/L leachates. The TOC concentrations in the pretreatment leachates also increased, from 30 to 190 mg/L, as the solid-to-liquid ratio increased. Oil and grease increased from 4.4 mg/L in the 1:100 S/L leachates to 16 mg/L in the 1:4 S/L leachates. PCBs were not detected in any of the pretreatment leachates.

Six metals were observed in the pretreatment BET leachates. Most of these increased in concentration as the solid-to-liquid ratios increased from 1:100 to 1:4. Metals exhibiting this trend were arsenic (0.19 to 0.38 mg/L), calcium (19 to 150 mg/L), and barium (0.023 to 0.11 mg/L). Aluminum, cadmium, and sodium were present at detection limit concentrations in one or more of the leachates. Lead was not detected in the pretreatment BET leachates.

BET leachates from the post-treatment Off-Site Area One wastes had pH values ranging from 11.8 to 12.1. The Ehs ranged from -77 mv in the 1:4 S/L leachates to -50 in the 1:100 S/L leachates. The TDS in the post-treatment leachates increased from 980 mg/L in the 1:100 S/L leachates to 4600 mg/L in the 1:4 S/L leachates. The TOC concentrations in the post-treatment leachates also increased, from 14 to 120 mg/L, as the solid-to-liquid ratio increased. Oil and grease concentrations increased from 3.7 mg/L in the 1:100 S/L leachates to 26 mg/L in the 1:4 S/L leachates. PCBs were not detected in any of the post-treatment leachates.

Nine metals were observed in the post-treatment BET leachates. Most of these increased in concentration as the solid-to-liquid ratios increased from 1:100 to 1:4. Metals exhibiting this trend were arsenic (0.0097 to 0.067 mg/L), copper (0.020 to 0.17 mg/L), barium (1.4 to 9.7 mg/L), calcium (410 to 1,000 mg/L), and sodium (4.0 to 80 mg/L). Aluminum decreased from 0.83 mg/L in the 1:100 S/L leachate to <0.20 mg/L in the 1:4 S/L leachate. Selenium, lead, and nickel were present at detection limit concentrations in one or more of the post-treatment BET leachates. Zinc was not detected in the post-treatment leachates.

7.3.4 ANS 16.1

The modified American Nuclear Society 16.1 (ANS 16.1) leaching test was performed on three replicate waste samples of the three treated wastes. This test simulates leaching from the intact (not crushed) treated waste in contact with rapidly flowing ground water by using a static sequential leaching method. Leachates were collected at 1, 3, 7, 14, and 28 days during the leaching test. All of the post-treatment samples maintained their structural integrity throughout the test period. No contaminants of concern were found in the ANS 16.1 leachates in concentrations sufficient to allow calculation of a leachability index, as described in the ANS 16.1 procedure. The ANS 16.1 leachate data are summarized in Tables 23 through 25. The detailed data are presented given in Appendix A, Tables A.30 through A.43.

TABLE 23

CHEMICAL ANALYSES OF ANS 16.1 LEACHATE FROM TREATED FILTER CAKE WASTE

	DAY 1	DAY 3	DAY 7	DAY 14	DAY 28
PCBs (μg/L)					
Aroclor-1242	<0.11	<0.10	<0.11	<0.020	<0.11
Aroclor-1260	<0.21	<0.21	<0.22	<0.040	<0.22
Metals (AA) (mg/L)					
Arsenic	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mercury	<0.00030	<0.00020	<0.00030	<0.00020	<0.00020
Selenium	<0.0040	<0.0040	<0.0050	<0.0040	<0.0040
Thallium	<0.0020	<0.0020	<0.0020	<0.0010	<0.0020
Metals (ICPES) (mg/L)					
Aluminum	<0.20	0.27	0.30	<0.20	0.37
Barium	0.17	0.19	0.22	0.25	0.28
Beryllium	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cadmium	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
Calcium	63	63	72	81	100
Chromium	<0.030	<0.030	<0.030	<0.030	<0.030
Copper	<0.020	<0.020	<0.020	<0.020	<0.020
Lead	<0.050	<0.050	<0.050	<0.050	<0.050
Nickel	<0.020	<0.020	<0.020	<0.020	<0.020
Sodium	7.3	5.2	4.4	5.3	5.0
Zinc	<0.020	<0.020	<0.020	<0.020	<0.020
Other Chemical Tests					
Eh (mV)	-20	-15	-36	-41	-57
Filterable Residue (TDS) (mg/L)	310	270	310	340	490
Oil & Grease, infrared (mg/L)	<0.40	<0.40	<0.50	<0.40	<0.40
pH (pH units)	10.7	10.9	11.0	10.7	11.3
Total Organic Carbon (mg/L)	6.6	5.2	5.3	6.3	7.0

TABLE 24

CHEMICAL ANALYSES OF ANS 16.1 LEACHATE FROM TREATED FILTER CAKE/OILY SLUDGE MIXTURE

	DAY 1	DAY 3	DAY 7	DAY 14	DAY 28
PCBs (μg/L)					
Aroclor-1242	<0.11	<0.10	<0.11	<0.020	<0.10
Aroclor-1260	<0.21	<0.20	<0.22	<0.040	<0.20
Metals (AA) (mg/L)					
Arsenic	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mercury	<0.00020	<0.00020	<0.00020	<0.00020	<0.00020
Selenium	<0.0040	<0.0040	<0.0050	<0.0040	<0.0040
Thallium	<0.0020	<0.0020	<0.0020	<0.0010	<0.0020
Metals (ICPES) (mg/L)					
Aluminum	0.57	0.57	0.53	0.50	0.70
Barium	0.32	0.35	0.37	0.39	0.40
Beryllium	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cadmium	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
Calcium	93	95	98	93	88
Chromium	<0.030	<0.030	<0.030	<0.030	<0.030
Copper	<0.020	<0.020	<0.020	<0.020	<0.020
Lead	<0.050	<0.050	<0.050	<0.050	<0.050
Nickel	<0.020	<0.020	<0.020	<0.020	<0.020
Sodium	17	11	9.8	12	16
Zinc	<0.020	<0.020	0.037	<0.020	<0.020
Other Chemical Tests					
Eh (mV)	-24	-22	-33	-52	-62
Filterable Residue (TDS) (mg/L)	380	310	340	350	340
Oil & Grease, infrared (mg/L)	<0.40	<0.40	<0.40	<0.50	<0.40
pH (pH units)	11.1	11.1	11.2	10.9	11.3
Total Organic Carbon (mg/L)	6.3	5.3	5.3	5.3	6.0

TABLE 25

CHEMICAL ANALYSES OF ANS 16.1 LEACHATE FROM TREATED OFF-SITE AREA ONE WASTE

	DAY 1	DAY 3	DAY 7	DAY 14	DAY 28
PCBs (μg/L)					
Aroclor-1242	<0.21	<0.21	<0.11	<0.020	<0.11
Aroclor-1260	<0.42	<0.42	<0.22	<0.040	<0.22
Metals (AA) (mg/L)					
Arsenic	0.0070	0.0053	0.0063	0.0063	0.0080
Mercury	<0.00020	<0.00020	<0.00020	<0.00030	<0.00020
Selenium	<0.0040	<0.0040	<0.0050	<0.0040	<0.0040
Thallium	<0.0020	<0.0020	<0.0020	<0.0010	<0.0020
Metals (ICPES) (mg/L)					
Aluminum	<0.20	0.30	0.37	0.43	0.73
Barium	0.33	0.42	0.54	0.67	0.90
Beryllium	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cadmium	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
Calcium	110	130	150	170	220
Chromium	<0.030	<0.030	<0.030	<0.030	<0.030
Copper	<0.020	<0.020	<0.020	<0.020	<0.020
Lead	<0.050	<0.050	<0.050	<0.050	<0.050
Nickel	<0.020	<0.020	<0.020	<0.020	<0.020
Sodium	17	11	9.9	11	13
Zinc	<0.020	<0.020	<0.020	<0.020	<0.020
Other Chemical Tests					
Eh (mV)	-32	-28	-48	-67	-78
Filterable Residue (TDS) (mg/L)	610	570	620	740	870
Oil & Grease, infrared (mg/L)	1.9	1.7	1.9	1.1	3.2
pH (pH units)	11.1	11.4	11.4	11.1	11.7
Total Organic Carbon (mg/L)	13	11	13	11	20

7.3.4.1 Filter Cake

The pH remained essentially constant throughout the leach test, ranging from 10.7 to 11.3 pH units. The Eh also exhibited little change over the test period, increasing from about -15 to -57 mv. Oil and grease was not found in any of the ANS 16.1 leachates generated from the post-treatment filter cake wastes. The total organic carbon remained essentially constant (5.2 to 7.0 mg/L) over the test period. The total dissolved solids of the post-treatment leachates ranged from 270 to 490 mg/L.

PCBs were not detected in the leachates. The only metals detected in the leachates were aluminum (<0.20 to 0.37 mg/L), barium (0.17 to 0.28 mg/L), calcium (63 to 100 mg/L), and sodium (4.4 to 7.3 mg/L). Silver and lead were each found at detection limit concentrations in only one of the fifteen ANS 16.1 leachates analyzed.

7.3.4.2 Filter Cake/Oily Sludge

The pH of the filter cake/oily sludge ANS 16.1 leachates ranged from 10.9 to 11.3. The Eh ranged from -24 mv in the day 1 leachates to -62 mv in the day 28 leachates. Oil and grease was not detected in any of the ANS 16.1 leachates. The total organic carbon and the total dissolved solid remained essentially constant over the test period, ranging from 5.3 mg/L to 6.3 mg/L and 310 mg/L to 380 mg/L, respectively.

PCBs were not detected in the filter cake/oily sludge ANS 16.1 leachates. Aluminum (0.50 to 0.70 mg/L), barium (0.32 to 0.40 mg/L), calcium (88 to 98 mg/L), and sodium (9.8 to 17 mg/L) were found in the ANS 16.1 leachates. Silver, thallium and zinc were found at detection limit concentrations in one of more of the leachates.

7.3.4.3 Off-Site Area One

The pH of the Off-Site Area One ANS 16.1 leachates ranged from 11.1 to 11.7. The Eh values ranged from -28 mv to -78 mv. The oil and grease content of the leachate ranged from 1.1 mg/L to 3.2 mg/L. The TOC varied from 11 mg/L to 20 mg/L. Total dissolved solids increased from 570 mg/L to 870 mg/L.

PCBs were not detected in the Off-Site Area One ANS 16.1 leachates. Barium (0.30 to 0.90 mg/L) and calcium (110 to 220 mg/L), increased slightly throughout the leach test. Sodium

(9.9 to 17 mg/L) and arsenic (0.0053 to 0.0080 mg/L) remained relatively constant in all 15 leachates.

7.4 MORPHOLOGICAL TESTS

The Demonstration Plan provides for periodic petrographic examination of cement-solidified waste samples over the next several years. Data from these analyses will determine the extent to which several routine characteristics of structural concrete are present or change:

- Adequacy of curing
- Degree of carbonation
- Degree of hydration and nature of hydration products
- Weather effects on the integrity of the cemented mass
- Degree of mixing of waste with cementitious materials

At the field demonstration, cast cylinder molds of each of the three wastes treated, plus molds of the sand reagent blank, were prepared. The absence of reliable published methods for petrographic examination and sample preparation of cement-solidified oily waste required in-depth consultation with experts in this field. As a result, an array of laboratory tests was devised, to be conducted by both U.S. EPA contractor laboratories and the U.S. Army Corps of Engineers Waterways Experiment Station (WES). These tests include:

- X-ray radiography of cast cylinders (of cement-solidified waste)
- Sonic pulse analysis of cast cylinders
- Void characterization on polished surface of cast cylinders
- Preparation and examination of thin-sections of cast cylinders
- X-ray diffraction analysis of minerals in cast cylinders
- Electron microscope examination

Three cylinders of each of the post-treatment filter cake, filter cake/oily sludge, Off-Site Area One, and the reagent mix/control samples were initially submitted for X-ray radiographic analyses. At the time of preparation of this report, only preliminary X-ray radiographs of these samples had been completed. These clearly showed numerous small round spots throughout the three cylinders photographed in preliminary work. Since the photographs were at scale, it could be seen that the observed spots corresponded, both in approximate size and distribution, to the dark oily spots observed in the treated waste monoliths (see Section 7.5). These early observations lend credibility to the representativeness of cast cylinders to the larger treated waste monoliths.

Each of the twelve cylinders mentioned above will also be subjected to sonic pulse analysis. This test will be used to assess the long-term stability of the cylinders. Four of these cylinders, one from each area plus a reagent mix, will be retained for periodic long-term analysis.

Longitudinal slabs approximately 75 x 150 mm in size and 20 mm in thickness will be cut from the above cylinders. One slab will be prepared from the reagent mix. One slab will be prepared from each of the two cylinders for each treatment area. Slab preparation will entail cutting and then polishing one surface of each slab.

The polished slabs will be analyzed to determine the distribution of air and waste-filled voids in the surface of the slabs. These slabs will then be analyzed to assess incorporation of oily material by a technique that measures ultraviolet reflectance on the polished surface. X-ray radiographic analysis will also be performed on the slabs. Point counts of voids will be performed using a stereoscopic microscope and computerized image analysis techniques. The prepared slabs will also be photographed.

Scanning electron microscope (SEM) analysis will be conducted to determine morphological features of the solidified matrix under moderately high magnification (100x-1000x). This analysis will include visual interpretation of the slab surface as well as a determination of the distribution of diatomaceous earth in cylinders containing the filter cake waste.

X-ray diffraction (XRD) analysis will be conducted to determine the presence of crystalline material in the solid waste matrix. The XRD analysis will also be used to help identify the types of cementitious hydration products in the solidified materials.

Methods for preparing thin sections of the solidified wastes will be investigated. This task is complicated by the presence of oil in the solidified waste, as cutting the thin sections is expected to disturb the oil. After the sections have been prepared, they will be analyzed and photographed using a petrographic microscope with transmitted light.

7.5 LONG-TERM TESTS

This section discusses the planned long-term tests on the treated waste materials from the Soliditech demonstration. These include the Waste Interface Leaching Test (WILT), TCLP and EP extraction tests, and physical stability testing of the treated waste material.

7.5.1 Waste Interface Leaching Test

The purpose of the WILT is to determine if the Soliditech process will reduce the long-term leachability of the waste to safe levels. The WILT consists of two sizes of solidified cylinders (3"d x 18"h and 6"d x 18"h) from each of three solidified wastes (Off-Site Area One, filter cake, and filter cake/oily sludge). Leachate will be collected and analyzed from the WILT on a biweekly basis for a period of one year. The first 16 weeks of WILT results are contained as preliminary data in Appendix G of this report. Each of three replicate cores of each type of waste was placed in an individual HDPE tank and the annular space between the core and the container was filled with acid-washed sand. A 1-inch layer of sand was added to the top of the column to disperse the leaching fluid (distilled water) uniformly over the cross-sectional area of the waste core. Leachate from each column was collected in a Tedlar® bag fitted to the base of the tank. Distilled water was added to saturate each column to the surface level of each column. Each column remained saturated over the two-week interval prior to sampling. The leachates were analyzed for pH, Eh, metals, and PCBs.

PCBs, lead, and zinc were not detected in the WILT leachates from the solidified wastes. The cumulative amounts (mass/cm^2) of aluminum, barium, calcium, TOC, and TDS leached from the solidified wastes over a 16 week period are shown in Table 26. TSD concentrations leached from the solidified wastes, which are indicative of the porosity of the solidified wastes, were lowest from the filter cake/oily sludge mixture. These results are supported by the physical test data which indicate that the filter cake/oily sludge mixture is the least permeable of the solidified wastes (Table 14). Concentrations of metals and organics in leachate from the WILT are reported in Appendix G. All elemental and organic concentrations were below levels of concern for protection of groundwater quality.

7.5.2 TCLP and EP Extracts of Solidified Waste

TCLP and EP tests will be performed on molded waste samples from three types of waste (Off-Site Area One soil, filter cake, and filter cake/oily sludge mixture). Aging periods for the molded waste samples will consist of 28 days, 6 months, and 1, 2, and 5 years. Three replicate

TABLE 26

WILT TEST RESULTS THROUGH WEEK 16

	Off-Site Area One		Filter Cake		Filter Cake/ Oily Sludge	
	Column		Column		Column	
Parameter	Small	Large	Small	Large	Small	Large
pH	12.1	11.5	11.4	11.3	11.6	11.4
PCBs[a] (μg/cm^2)	ND[b]	ND	ND	ND	ND	ND
Metals[a] (μg/cm^2)						
Aluminum	15	29	17	18	25	33
Barium	20	3.9	9.4	7.6	8.0	4.1
Calcium	3400	1100	2680	2640	1380	930
Lead	ND	ND	ND	ND	ND	ND
TOC[a] (μg/cm^2)	NC[c]	520	ND	446	NC	147
TDS[a] (mg/cm^2)	NC	13.5	NC	15.5	NC	6.3

Notes:

[a] Cumulative amount leached from cylinders over 16 weeks is expressed as mass per cm^2 of cylinder surface area. The small cylinders are 3 inches in diameter and 18 inches in height. The large cylinders are 6 inches in diameter and 18 inches in height.

[b] Not detected.

[c] Not calculable.

samples of each waste type will be extracted at each time period. For each of these cases, a single core will be used for TCLP and EP extractions, resulting in a total of 60 cores over the entire test period.

Results from the TCLP and EP tests will provide data on the relative concentration of contaminants in extracts over a period of five years. These results will indicate any changes in extract toxicity that may be attributed to aging or weathering of the solidified waste.

7.5.3 Physical Stability of Treated Waste Monoliths

Initial observations indicate that, of the following characteristics, some will be easier to assess than others. Some features may be disregarded or others added at future observation points.

7.5.3.1 Surface Spalling

Upon removal of the plywood sides of the molds, a thin, dusty patina was present on the surface of the treated waste monoliths. An imprint of the texture of the plywood surface was obvious in this patina. This feature appeared to be a few millimeters in thickness and was easily removed with an armstrong wire brush. The presence of this material at the interface with the plywood container emphasizes the need to be cautious in describing monolith properties without first being sure that the container or other accessory to this demonstration does not contribute artifacts.

Block 1, containing the reagent mix or control sample, appeared to be similar to a competent commercial concrete, with no particularly notable features. All the blocks with waste in the composition had a thin veneer that appeared to form from splashing onto the inside of the plywood form when the fluid mix was discharged into each form. This material was easily chipped off with light hammer blows after the plywood forms were removed. Easily removed thin layers of solidified waste were hand-chipped off the top edges of all blocks prior to stacking for long-term evaluation.

Blocks 2, 3, and 4 contained black masses, several centimeters in dimension, over areas of tens of centimeters, on most faces of these blocks. These masses appeared to consist of relatively unmixed waste material. This was the first waste batch mixed, and thorough mixing may not have been achieved in this batch. Observations at the next time interval should include note of these masses.

Blocks 5 - 10 contained no particular or notable features. The solidified waste appeared uniform as a blocky mass.

Blocks 11 - 14 also contained no particular or notable features.

7.5.3.2 Grain Exfoliation

No initially notable grain looseness was noted on any of the blocks except for the dusty surface adjacent to the plywood form sides. As noted previously, this was easily removed with a hand-operated wire brush, into a light dusty form. Other than occasional brushing on a few blocks, no attempt was made to remove this surface material.

7.5.3.3 Crack and Fissure Development

Except for Block 1, nearly all blocks containing waste exhibited circular cracks near the corners of the top surface. These are believed to be normal stress-relief cracks resulting from the use of square box forms. These weaknesses suggest that round forms be used in any subsequent construction of treated waste monoliths. Blocks 3, 5, and 11 exhibited these corner cracks one day after pouring the fluid waste, with Block 3 being most pronounced.

The northeast corner of Block 5 exhibited a severe crack with separation of a triangular corner volume approximately 6" in dimension and at least 12" length after Block 10 was placed upon it in the long-term stack.

No remarkable cracks or fissures were seen on any of the faces of the blocks after removing the plywood forms.

7.5.3.4 Oxidative Discoloration

No clear indications of oxidative processes were noted either after initial setting of the cement-solidified waste or after the 28-day curing period. A lightening of color of the blocks was noted within fractions of an hour after the plywood forms were removed. This was attributed to air-drying of the block surfaces.

Blocks 5 through 10, comprised of the solidified filter-cake waste pile material, were the darkest in overall color. Blocks 2 through 4 contained large black masses visible on all faces; as indicated earlier, these appeared to be unmixed masses of oily material.

On many of the blocks, where the plywood-surface-influenced finish of block faces or corners were chipped away, numerous rounded black spots were obvious. These appeared to be oil- or grease-filled voids; or alternatively, small volumes of oily waste surrounded by the cementitious matrix. The spots varied in size up to several millimeters. Some spots were surrounded by a "halo", indicating that liquid or volatile components of the oily matrix had migrated radially outward from the original globule. These halos ranged both in clarity and size, up to a few centimeters in diameter. At the time the blocks were stacked for long-term evaluation, the texture and composition of the black spots were not probed or assayed in the field, nor were samples specifically taken for detailed assay. These features will be examined as part of the long-term monitoring study.

7.5.3.5 Salt Efflorescence

A significant accumulation of white salt was observed on the top surface of Block 8. This accumulation was present in the concave depressions remnant from the removal of sample material from the block surface after the fluid mix was discharged from the mixer. Although other blocks may have contained salt accumulations, none was as concentrated as that of Block 8. This material was undisturbed and not sampled at the time the blocks were stacked for long-term evaluation. This material will be examined and sampled during long-term monitoring activities.

7.5.3.6 Pore Characterization

This parameter is vividly illustrated by the presence of the black spots noted previously. Initial conclusions represent these spots as oil- or grease-filled voids or pores. This must be verified by laboratory examination of similar features in the cast cylinders taken for laboratory study. This detailed examination should determine whether the black spots are indeed filled voids or waste globules surrounded by cementitious matrix. No clearly visible air voids could be seen in the treated waste monoliths. Planar cracks of various sizes were occasionally visible in some blocks. The major cracks were described earlier.

7.6 MATERIALS BALANCE

Table 27 presents material balance information for each of the four treatment runs performed by Soliditech during the demonstration. The data in this table are based upon information collected during the demonstration by Soliditech (Soliditech, 1989a). Table 28 gives the observed volumes and weights of the treated waste monoliths.

TABLE 27

MATERIALS BALANCE

Estimated Weights of Materials in Pounds

	Reagent Mixture	Filter Cake/ Oily Sludge	Filter Cake	Off-Site Area
Waste Material	800(a)	3,950	11,200	9,100
Volume (yd^3)	0.24	1.94	5.83	4.28
Type II Cement(b)	442	4,970	4,920	4,540
Urrichem	8.96	39.2	111	90.7
Additives	16.5	140	167	136
Water	154	666	2,687	1,830
TOTAL ESTIMATE WEIGHT	1,420	9,760	19,100	15,700
	Measured Weights of Products in Pounds			
Material in Large Forms	1,250	8,410	15,400	10,900
Laboratory Samples	31.0	305	266	291
Field Tests	0	144	188	206
Residual(c)	50	250	250	---(d)
Screened Material(e)	---	---	---	300
TOTAL MEASURED WT.	1,330	9,110	16,100	11,700
Volume (yd^3)(f)	0.455	3.08	6.25	4.10
Weight Differential	90	650	3,000	4,000
Percent Weight Difference(g)	6.7	7.1	18.6	34.2

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[a] Clean sand used rather than waste material for this test run.

[b] According to Soliditech, fluffing of the cement may cause the cement weight values to be as much as 5 percent high.

[c] According to Soliditech, the residual in the mixer after treatment might be as much as 1/10 cubic-yard, or approximately 250 pounds in the large mixer and approximately 50 pounds in the small mixer. We will assume the maximum amount for each test run. The mixer was not cleaned prior to treating the Off-Site Area One waste, so no correction was made to that value.

[d] The residual of the preceding test run was allowed to remain in the mixer and was treated with this test run. It was assumed that the residual weight was equal for each of these test runs. Thus, the amount of residual remaining in the mixer after treatment should equal the amount of residual in the mixer prior to treatment and no correction was needed.

[e] Approximate amount of material removed by screening according to Soliditech.

[f] The total cubic-yards measured plus volume of treated material collected for laboratory and field experiments.

[g] Weight Differential divided by Total Measured Weight times 100.

TABLE 28

OBSERVED VOLUME AND WEIGHT OF TREATED MATERIAL

Plywood Form Number(a)	Volume of Material(b)	Net Weight of Material(c) Before Cure	After Cure
Reagent Mix/Control			
1	0.444	1248	1258
Oily Sludge/Filter Cake			
2	1.10	3116	3080
3	1.08	3228	3186
4	0.722	2070	2062
Filter cake			
5	1.00	2512	2484
6	1.04	2700	2668
7	1.14	2958	2934
8	0.972	2380	2354
9	0.972	2432	2406
10	0.806	2026	2004
10a	0.136	342	374
Off-Site Area One Soil			
11	0.972	2644	2608
12	1.08	3034	3000
13	1.06	2940	2910
14	0.806	2256	2240
TOTALS	13.3	35,886	35,700

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[a] See Section 6.3.2 for a description of the treated waste material contained in each form.

[b] Volume is expressed in cubic-yards.

[c] Net weight is expressed in pounds. These weights were obtained by weighing the treated waste monoliths both before and after the 28 day curing period. No attempt was made to explain the small variations in the two field weights.

The waste content of the treated material ranged from 40 to 59 percent by weight, with an average of 52 percent. The volume increase of the waste due to treatment ranged from no increase to 59 percent increase, with an average of 22 percent. The bulk density of the treated waste increased by 25 to 41 percent, with a 31 percent average.

It should be noted that the weights of the ingredients for each of the four runs were found to be greater in sum than the weight of the treated material. This can be attributed to several factors. The amount of waste material treated in each of the waste runs, particularly the Off-Site Area One soil, was probably overestimated. The actual weight of cement added to each mixture may also be overestimated by as much as 20 percent. It is unlikely that the weight of the water, Urrichem, or additives is significantly different than that reported, because these ingredients were either carefully measured or metered into the mixture.

7.7 CONCLUSIONS

The unconfined compressive strength values, very low permeability, and high resistance to wet/dry and freeze/thaw deterioration demonstrates a high degree of physical stability of the three treated wastes. Although the treated waste had a higher bulk density than the raw waste, a small volume increase accompanied the solidification/stabilization process.

The concentrations of all contaminants found in the EP and TCLP extracts of treated samples are low. It is significant that lead, as measured by TCLP, EP, ANS 16.1, and WILT procedures, is barely detectable in extracts of treated wastes. This indicates a high degree of stability in the solidified/stabilized matrix of treated waste. The BET data confirm the stability of the treated wastes against leaching loss of lead and arsenic. The low amounts of contaminant solutes found in the preliminary WILT leachates confirm the parallel findings in the TCLP, EP, BET, and ANS 16.1 extraction tests.

Measurable amounts of arsenic, barium, chromium, copper, lead, nickel, and zinc appeared in the treated wastes. The source of these elements is believed to be portland cement. Decreases in loss-on-ignition are most likely due to dilution by the added cement.

The absence of any mechanical equipment problems during the demonstration illustrated the reliability of the Soliditech system. After the equipment operator gained familiarity with waste materials at this site, the process mixed all components into a homogenous solidified product.

8.0 COST OF DEMONSTRATION

The cost of demonstrating Soliditech's solidification/stabilization process at the Imperial Oil site was approximately $958,300. This cost includes site characterization and preparation, demonstration planning and field work, chemical analyses, and report preparation. The developer's portion of this cost was $232,600. The balance of $725,700 was allocated to the U.S. EPA SITE Program.

8.1 U.S. EPA SITE CONTRACTOR COSTS

Technical support to the U.S. EPA SITE program for the evaluation of the Soliditech technology was provided by a contractor. Each SITE project is divided into two phases -- planning (Phase I) and demonstration (Phase II). Phase I costs are actual costs; Phase II costs include actual costs plus labor estimates through the completion of report preparation. Specific activities under each phase and a cost breakdown for each phase are presented below (all costs are rounded to the nearest $100).

8.1.1 Phase I: Planning Stage

Phase I activities included:

- **Solidification/stabilization technology review**
- **Protocol evaluation**
- **Site sampling and treatability testing**
- **Development of the demonstration plan**
- **Site subcontractor procurement**

Costs for Phase I activities are summarized below:

• **Labor**	**$ 82,200**
• **Equipment and supplies**	**9,300**
• **Travel**	**8,300**
• **Chemical analyses**	**29,200**

8.1.2 Phase II: Demonstration Stage

Phase II activities included:

- Mobilization and site preparation
- Sample collection and field oversight
- Sample processing after the 28-day curing period
- Chemical analyses
- Report preparation

Costs for Phase II activities are summarized below:

• Labor	$ 244,100
• Equipment and supplies	63,400
• Travel/transportation	21,400
• Chemical analyses	261,900
• Miscellaneous	5,900

Labor costs include estimates through report preparation. Miscellaneous costs include the demonstration area security and the decontamination facility. Transportation costs include equipment mobilization and demobilization.

8.2 DEVELOPER (SOLIDITECH) COSTS

The costs presented in this section were provided by Soliditech (Soliditech, 1989b). These costs were based upon all expenses incurred by Soliditech in preparing for and conducting the SITE demonstration.

• Labor	$ 21,600
• Laboratory	7,400
• Travel	10,600
• Equipment	177,000
• Transportation	13,000
• Raw materials	1,300
• Miscellaneous	1,700

Equipment and miscellaneous costs include such one-time costs as health and safety equipment and training. Raw materials costs include pozzolan (portland cement), Urrichem, and other proprietary additives. Transportation costs include equipment mobilization and demobilization.

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